A Summary of Current Program, 7/1/66

and Preliminary Report of Progress

for 7/1/65 to 6/30/66

SOUTHERN UTILIZATION RESEARCH AND

DEVELOPMENT DIVISION

of the

AGRICULTURAL RESEARCH SERVICE

UNITED STATES DEPARTMENT OF AGRICULTURE

and related work of the

STATE AGRICULTURAL EXPERIMENT STATIONS

This progress report of U.S.D.A. and cooperative research is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

The summaries of progress of U.S.D.A. and cooperative research include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed, will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of U.S.D.A. and cooperative research issued between July 1, 1965 and June 30, 1966. Current agricultural research findings are also published in the monthly U.S.D.A. publication, Agricultural Research. This progress report was compiled in the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, New Orleans 70124, Louisiana.

UNITED STATES DEPARTMENT OF AGRICULTURE
Washington, D. C.
July 1, 1966

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INTRODUCTION

The program of the Southern Utilization Research and Development Division is an organized effort through science and technology to increase present uses and to discover and develop varied new uses for Southern farm crops. Our farmers need new markets and strengthened demand for their production. At the same time, the Nation needs the new and better products that science can create from agricultural materials. To this end the Division conducts research on cotton, cottonseed, peanuts, citrus and subtropical fruits, peaches, rice, sweet sorghum, pine gum, sweetpotatoes, cucumbers, and other vegetables.

The Division's program includes basic and applied research in the physical and biological sciences and engineering. Basic research plays a key role in uncovering new information that may be later exploited in applied research and development. When appropriate, engineers carry out pilot-plant studies of promising laboratory developments to provide engineering and cost data essential to industrial application feasibility determinations. The Southern Division has a total staff of about 565 and the in-house scientific effort in its research program amounts to approximately 187 scientific man-years. The Division consists of two Pioneering Research Laboratories (Seed Protein and Plant Fibers), eight commodity-oriented Laboratories (Cotton Finishes, Cotton Chemical Reactions, Cotton Mechanical, Cotton Physical Properties, Oilseed Crops, Food Crops, Fruit and Vegetable Products, and Naval Stores), and one Laboratory (Engineering and Development) for engineering research and development. Headquarters of the Southern Division are located at the Southern Regional Research Laboratory, New Orleans, Louisiana. The Division also has personnel and laboratory facilities at Winter Haven and Olustee, Florida; Weslaco, Texas; Raleigh, North Carolina; and Natick, Massachusetts.

Division scientists consult with specialists from other organizations during both the planning and the execution of the research, and cooperate actively with industry to facilitate commercialization and utilization of new findings. Much of the cooperation is informal, but some work is conducted under conditions described in written cooperative agreements and memorandums of understanding. Currently 64 such agreements are in effect.

The farm products with which the Southern Division deals not only provide food, clothing and industrial raw materials, but also contribute to the Nation's general prosperity and well-being. Cotton, the Nation's number one cash crop, has an annual farm value of about $2.2 billion. The retail value of cotton products is almost $16 billion. Cottonseed, a byproduct of cotton, has a farm value of around $290 million. The retail value of its products is about $1.1 billion. Citrus grown in the U.S. has a farm value of over $560 million; vegetables $1.3 billion; peanuts almost $285 million; rice about $375 million; and pine gum about $20 million. Industries processing these agricultural crops play a vital role in the Nation's economy; agri-business today is about 40% of the Nation's total economy.

There is an urgent need to maintain the traditional food, feed and industrial outlets for agricultural products and to create new and larger markets for them. Utilization research is needed to solve existing problems, to permit adjustment to important trends and to develop entirely novel operations. The opportunities are great and, as shown by past experience, the ability of utilization research to benefit the economy is tremendous. Following are a few examples of significant developments based on the research of scientists at the Southern Division.

Examples of Recent Accomplishments of the Southern Utilization Research and Development Division

Improved All-Cotton Durably Pressed Garments Promise to Regain Markets for Cotton. Because the original all-cotton wash-wear, durably pressed garments had low abrasion resistance, blends containing 50 percent or more of the synthetics have largely replaced 100 percent cotton in these items, especially men's wear. However, intensive Department research has recently made impressive progress in improving the performance of all-cotton garments, thereby improving cotton's competitive position in this important large-volume market. Several ways have been found for improving the resistance of the cotton garments to abrasion during wearing, laundering, and drying. They include proper selection of fabric; pretreatments such as slack mercerization; slack mercerization followed by stretching, especially of fabrics containing plied yarns; use of proper amount of crosslinking agent and avoiding of overcuring; use of polymers to coat fibers within the fabric structure, especially polymers that can be further polymerized or crosslinked; preferential crosslinking in selected regions of the fabric structure; use of selected polymers in conjunction with preferential crosslinking; vapor phase treatment of fabrics and garments with crosslinking agents and polymers; and blending of crosslinked cotton fibers with noncrosslinked fibers. By using certain combinations of two or more of these approaches, more than a twofold improvement in wear life has already been obtained. Present emphasis in the research is on the refinement of individual treatments and the establishment of optimum combinations to give maximum abrasion resistance and wear life. Industry is evaluating some of the more promising research findings.

New Chemically Treated Cotton Batting Enthusiastically Accepted by Industry. Industrial firms have been rapidly going into the production and utilization of Cotton Flote, the outstanding cotton batting product developed by Department scientists in cooperation with the Textile Waste Association, The National Cottonseed Products Association, The National Cotton Batting Institute, and the Foundation for Cotton Research and Education of the National Cotton Council. At least twelve companies are either producing the new chemically treated batting or installing the process in their plants. Two major U. S. automobile manufacturers began using Cotton Flote in some models of their 1965 and 1966 cars, and a third auto manufacturer has tentatively approved the product as an alternate for other padding materials currently in use. One bedding company is now

using Cotton Flote in high-grade mattresses. Firms in many foreign countries, including Germany, Holland, England, Canada, Pakistan, India, Australia, Thailand, Mexico, Switzerland, Japan, and Ireland, have also shown interest in the new process and product. Cotton Flote appears to have excellent potential for further growth in cushioning markets in the automotive, furniture, and bedding industries, markets which represent a potential usage of 1 1/2 million bales of cotton linters and textile wastes annually.

Partially Defatted Peanuts Now Produced Commercially. Industry sources have been quoted as expecting partially defatted peanuts to set off "the biggest advance in peanut consumption since man cracked open his first peanut shell." Each partially defatted peanut, prepared by mechanical pressing and without the use of solvents, contains up to 57% fewer calories than does a conventionally roasted peanut. One major company reports successful distribution of the product in small flexible bags and soon plans to introduce larger packages. Another company is producing and selling pressed peanuts for use in the new product. Still another is conducting market tests to decide whether or not it should go into full-scale production and distribution. Several other firms have shown interest in the product. In addition to its consumer appeal as a delicious snack, the new product may be useful as an ingredient in other processed and convenience foods and also appears to have potential as a rich source of protein.

Promising New Type of Cottonseed Oil Emulsion for Intravenous Nutrition Undergoing Extensive Evaluation. A high-calorie, nourishing fat emulsion that shows promise for intravenous nutrition has been developed by Department Scientists. The research is supported by the Office of the Surgeon General and is conducted in cooperation with the U. S. Army Medical Research and Nutrition Laboratory. This physically stable emulsion made from cottonseed oil could help physicians overcome the basic problem of preventing weight loss in patients fed intravenously for prolonged periods. Its preparation was made possible by development of a more efficient chromatographic method for isolation of pure egg lecithin, which is used as sole emulsifier. A large batch (85 liters) of the emulsion has been prepared without difficulty, and about three times this quantity will soon be prepared by the Harvard School of Public Health for clinical evaluation in human beings. Although this will be the first clinical test of the latest fat emulsion formulation, long term use with laboratory animals has been successful, producing satisfactory weight gain with minimal side effects.

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As a step toward implementation of the recommendations for a National Program of Research for Agriculture made jointly by the Association of State Universities and Land Grant Colleges and the USDA, a section has been added to each of the Areas in this report. It comprises a list of

the related publications of the State Agricultural Experiment Stations in addition to those heretofore reported covering the results of USDA and cooperative research. In future years, it is anticipated that information will be available to permit reporting of achievements resulting from State research in a format comparable to the present reporting of the USDA and cooperative research.

Examples of Recent Accomplishments of the State Agricultural Experiment Stations

Gamma Radiation Offers Promise for Extending the Shelf Life of Citrus Fruit and Reducing Spoilage Losses. Use of gamma radiation treatments for extending shelf life of citrus fruits has been found beneficial by workers of the Florida Agricultural Experiment Station. These scientists point out that there are many causes for citrus fruit spoilage, including stem-end rot, green mold, black rot of oranges and anthracnose. Although citrus fruits are not as perishable as peaches, strawberries and certain other common fruits, a sizeable loss occurs in marketing channels with estimates indicating that from 1959-1963, the retail value of the annual fresh market spoilage losses in the United States for oranges was $8.4 million and for grapefruit $5.4 million. The workers state that, while organisms causing decay are inactivated by an adequate exposure to the gamma rays, it is necessary to limit doses so that only minor changes take place in the fruit's texture, flavor, color and respiration. They further point out that the several decay-causing organisms vary in radiation resistance. Thus, no single radiation dose can be stated that will be the minimum needed for protecting citrus fruit against spoilage.

By-Products of Florida Citrus Characterized. The rapid growth in citrus production and processing has brought about a citrus by-products industry. Florida station scientists have developed information on their composition, technology and utilization. For example, citrus molasses which finds its greatest market presently as a feed, as now manufactured in Florida, is required to meet minimum State standards. It must contain 45 percent total sugars, expressed as invert sugar and have a Brix of not less than 35.5° by double dilution. This is typical of the wealth of valuable information compiled for the industry by these workers.

Packaged Fruit Products by Pasteurization-Refrigeration. Research in progress at the Alabama station has resulted in a new peach product made by a special pasteurization-refrigeration process that preserves the high qualities of fresh ripe peaches for a long period in packaged ready-to-serve form. The process involves packaging of sliced ripe peaches in glass jars with syrup, addition of vitamin C and citric acid to the pack, vacuum sealing, controlled pasteurization, chilling, and refrigerated (32-34° F.) storage. The pasteurization treatment is the key to high quality and long shelf life of the product. A special machine has been designed and a prototype model constructed to rotate the containers of peaches during pasteurization and cooling. A test pack of 13,000 cases has

been made in a commercial plant. More recent studies have been concerned with preservation of four additional fruits by the process. The process has been found effective in preserving the fresh qualities of ripe apples, pineapples, grapefruits and acidified cantaloupes.

Commercial Peanut Butter Ice Cream Developed. Research sponsored jointly by the Georgia Agricultural Experiment Station and the Georgia Agricultural Commodity Commission for Peanuts has resulted in the development of a formula and process for commercial peanut butter ice cream. Two commercial ice cream firms in Georgia cooperated in the development. Several firms outside the State evaluated it. Difficulties of moisture absorption and poor texture were overcome by very fine grinding and use of suitable stabilizers.

Drying Coffee with Solar Heated Air. The Puerto Rico Agricultural Experiment Station has demonstrated that by harnessing solar heat energy, substantial savings in electricity or fuel costs can be achieved in drying coffee. In some cases heating costs have been reduced by 70 percent. This is achieved by the construction of a solar heat collector on the roof of a processing building. Circulated air, heated in the collector is used in the drying process. This development will be of great value to coffee growers throughout the world since coffee is usually grown in areas where large quantities of solar energy are normally available.

"Instant" Pea and Bean Soups Being Perfected. "Instant" pea and bean soups are being perfected at Michigan State. Made from powdered peas and beans, these soups readily become wholesome, protein-packed foods just by adding water. Agricultural engineers and food scientists are trying to further improve the drying methods since the soups offer intriguing possibilities as inexpensive additions to the menus of underdeveloped countries.

High Temperature "Freezing" Gives Cheaper Fruit Juice. A faster and cheaper method of concentrating fruit juices is being explored by Wisconsin scientist O. R. Fennema. It involves freezing water at temperatures higher than 32° F. Certain compounds will make water molecules attract at temperatures higher than 32° F.--the normal freezing point of water. Freon 11, a common liquid in refrigerators, is such a compound. With the addition of Freon 11, apple juice, which under normal conditions freezes at 28° F., will freeze at temperatures about 15 degrees higher. Since only the water freezes, the unfrozen concentrated juice is separated by centrifugation. The Freon 11 is attached to the water removed from the juice and can be recovered and used again.

AREA 1 - COTTON UTILIZATION

Problem. Cotton, the nation's most important fiber crop, is facing severe and increasing competition from synthetic fibers. Cotton is America's largest source of cash farm income and still accounts for more than half of the total U. S. mill consumption of all major fibers. However, its proportionate share of the market has been slowly decreasing. The rapid growth of the synthetics at the expense of the natural fibers has been a phenomenon of the century. Expansion of market outlets for the chemical fibers has been based on vigorous research and development programs. The engineering and development programs of the chemical fiber industry are designed to capitalize on the special properties of each individual fiber as related to the functional use qualities desired in particular products; basically they involve the substitution of the newer fibers for cotton in cotton's traditional end-use markets. Expanded research to increase the utilization potential of cotton offers the most realistic opportunity for improving cotton's competitive strength as a textile fiber and for increasing cotton consumption. Basic studies of the chemical composition, physical properties and structure of cotton and modified cottons, chemical and physical investigations to improve cotton products, and research to develop the technology required for production of new and improved cotton products are basic to holding existing markets or expanding the use of cotton in new applications.

Fundamental information is badly needed in applied research to help cotton gain new and maintain old markets. Fundamental knowledge of the cotton fiber as to its structure, properties, and the mechanisms involved in chemical and physical behavior serves as a basis and a guide in the design and improvement of processing machinery, mechanical and chemical processes, and in the development of new and improved cotton yarns, fabrics, finishes, and treatments. Many chemical and physical treatments, as well as textile organizations and machine designs, offer a basis for the improvement of cotton quality or lowering of processing costs. Exploratory chemical and physical research is needed to determine the true potential of such approaches prior to undertaking extensive developmental research or the construction of prototype machinery. New or improved mechanical processing methods and textile machinery are urgently needed by the cotton industry. Cotton products with various special properties and processes for their production must likewise be developed to meet the serious competition of synthetic fibers and other competitive materials in numerous end uses. Some of the more important types of products that need to be developed or improved include: wash-wear textiles; wash-wear, durably pressed garments with adequate abrasion resistance; stretch and bulked products; weather- and rot-resistant fabrics; flame-resistant products; soil-resistant textiles; fabrics with water and/or oil repellency; textiles with improved luster; textiles with multifunctional properties; and insect-resistant bags for storage and shipment of food commodities. An essential part of such a utilization research program is

the development of new and improved methods and instruments for measuring the physical and chemical properties of cotton and its products.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, analytical chemists, physical chemists, physicists, microscopists, chemical engineers, mechanical engineers, statisticians, cotton technologists, textile technologists, and textile engineers engaged in both basic and applied research to help cotton gain new and maintain old markets. Cooperation is maintained with greige goods mills, textile mills, finishers, chemical manufacturers, manufacturers of textile machinery, cotton merchants, textile research institutes, and industry associations in connection with the research. The Crops Research Division, ARS, and the Cotton Division, C&MS, also cooperate, particularly in the procurement of cotton with special fiber properties and of known history. Cooperation is also maintained with the Market Quality Research Division, ARS, to insure coordination of effort in any related research.

Basic research on the chemical composition, physical properties and structure of cotton and its products is carried out at the Southern Regional Research Laboratory, New Orleans, Louisiana. This research provides information on the relationship of properties and structure to behavior of cotton in mechanical and chemical treatments, essential to an understanding of the performance of fibers during processing and in textile products. The development of new methods and instrumentation for measuring physical and chemical properties of cotton and its products is an integral part of the program. Also included is the research of the Plant Fibers Pioneering Research Laboratory to obtain basic information on the supermolecular structure of plant textile fibers; and to relate information of polymer and fiber structure to the mechanical and textile behavior of fibers. Additional basic research on chemical and physical properties and structure of cotton is being carried out: (1) under contracts at Stanford Research Institute, South Pasadena, California, on development of a method for counting neps in cotton at various stages of textile processing; and on development of a research instrument for accurately and automatically determining length, length distribution and diameter of cotton fibers; at Texas Agricultural Experiment Station, College Station, Texas, on the effect of variation in structure on cotton fiber properties caused by environmental and genetic factors; at the University of Tennessee, Knoxville, Tennessee, on investigations to determine the effects of fiber extensibility on fiber breakage in mechanical processing; at the Polytechnic Institute of Brooklyn, Brooklyn, N. Y., on relationship of molecular size, nature, shape, conformation, and configuration of organic nonaqueous compounds to their swelling power on cotton cellulose; and at Harris Research Laboratories, Inc., Washington, D. C., on investigation of factors influencing comfort in cotton apparel fabrics; and (2) under grants at Massachusetts Institute of Technology, Cambridge, Massachusetts, on investigation of fiber and yarn geometry in areas of deformation in cotton

fabrics; and at Georgia Tech Research Institute, Atlanta, Georgia, on elucidation of the role of fiber morphology on frictional behavior.

Chemical and physical investigations to improve products is also conducted at New Orleans, Louisiana. Exploratory chemical and physical research is carried out as a basis for improving mechanical and chemical processing, and developing new and improved yarns, fabrics, finishes, and treatments. One phase of the research--exploratory investigation of reversible chemical reactions to obtain information basic to the development of a commercially feasible reversible crosslink--is conducted with cooperation and support by the Cotton Producers Institute. The International Lead Zinc Research Organization cooperates in and supports exploratory research to impart useful properties to cotton through application of selected lead and other metal compounds. Additional exploratory chemical and physical investigations are also being carried out: (1) under contracts at Macrosonics Corporation, Carteret, New Jersey, on treatment of cotton fibers with acoustic energy; at Gagliardi Research Corporation, East Greenwich, Rhode Island, on chemical modification of cotton through treatments with reagents in the vapor phase; at Harris Research Laboratories, Inc., Washington, D. C., on the development of finishes for cotton fabrics to render them more rapid drying; and on the effect of soiling environment on the soiling tendency of various types of cotton finishes; at Southern Research Institute, Birmingham, Alabama, on the development of wash-wear cotton fabric with improved moisture absorptivity by use of reactive swelling agents; at Bjorksten Research Laboratories, Inc., Madison, Wisconsin, on the effect of resin thermoplasticity or thermosettability on the resistance of treated cotton fabrics to abrasion; and (2) under grants at Textile Research Institute, Princeton, New Jersey, on crosslinking of chemically modified cotton to obtain cotton fabrics with an optimum combination of resilience and thermoplasticity; and at the University of Arizona, Tucson, Arizona, on correlation of surface microtopography of treated and untreated cotton fibers with resistance to soiling of cotton textiles.

Research on process and product development is carried out at New Orleans, Louisiana. Major facets of the work include the development of: improved procedures for mechanical processing of cotton, new and improved textile machinery (opening through carding), and cotton products with special properties for various end uses. The principal types of products currently involved in the latter research include: wash-wear yard goods and garments, including those with durable press features; weather- and rot-resistant fabrics; soil-resistant textiles, including those with water and/or oil repellency; flame-resistant products; textiles with multifunctional properties; textiles with improved luster, strength and durability; stretch and bulked products; and insect-resistant bags. Pilot-plant evaluations of promising laboratory processes and products are carried out, and cost estimates are made to aid industrial establishment of various research developments. Close cooperation is maintained with cotton textile machine manufacturers and cotton textile processors in the evaluation of experimental textile machinery developed in the research, and in the establishment and

dissemination of engineering specifications for the commercialization of the new machinery. The research to develop cotton fabrics with improved resistance to outdoor weathering is cooperative with the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America) and the Canvas Products Association International. The Cotton Producers Institute cooperates in and supports research to develop optimal cotton fabric structures for men's trousers and dress suits. Research on cotton batting is conducted cooperatively with the National Cotton Batting Institute, Textile Waste Association, National Cottonseed Products Association and the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America). The Stored-Product Insects Research and Development Laboratory, Market Quality Research Division, ARS; bag manufacturers; and the Textile Bag Manufacturers Association cooperate in the work to develop improved insect-resistant cotton bags.

Additional research on process and product development is being carried out under contract at Auburn Research Foundation, Inc., Auburn, Alabama, to determine optimum processing procedures for cotton differing in tensile and elastic properties and relate these properties to mechanical processing performance, yarn, and fabric properties; at Georgia Tech Research Institute, Atlanta, Georgia, to develop improved cotton sewing thread for wash-wear fabric structures, compatible with existing high-speed manufacturing methods, which will not cause seam pucker, or which will have a markedly reduced tendency to cause seam pucker; at Texas Woman's University, Denton, Texas, on development of weather-resistant, water-repellent finishes for cotton; at Southern Research Institute, Birmingham, Alabama, on investigation of interfacial and graft polymerization procedures for producing weather-resistant cotton textiles with improved physical properties; at Fabric Research Laboratories, Inc., Dedham, Massachusetts, on development of improved coated cotton fabrics with optimum strength-weight characteristics for outdoor uses; at North Carolina State University, Raleigh, North Carolina, on evaluation of stretch-type cotton yarns (prepared by back-twisting and falsetwisting techniques) in knit wear; and on determination of optimum yarn constructions, knitting structures and prefabrication design for producing stretchable articles of knitted cotton wearing apparel by slack mercerization.

Other research on chemical and physical properties and structure of cotton is in progress under grants of P. L. 480 funds to the following foreign institutions: Swedish Institute for Textile Research, Gothenburg, Sweden, for an investigation of setting reactions in cotton fabrics (project duration - 5 yrs.); Fiber Research Institute, T.N.O., Delft, Holland, for an investigation of the fundamental mechanisms and bonding forces that could be used to improve tensile strength and other physical properties of cotton textiles (project duration - 5 yrs.); Ahmedabad Textile Industry's Research Association, Navrangpura, Ahmedabad, India, for a study of the relation between the fine structure and mechanical properties of cotton fibers by swelling and stretching treatments (project duration - 5 yrs.); and for a study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber (project duration -

5 yrs.); University of Bombay, Bombay, India, for an investigation of the photochemical degradation of cotton (project duration - 5 yrs.); and for an investigation of new solvents for molecular weight determination of cellulose (project duration - 3 yrs.); Indian Central Cotton Committee, Bombay, India, for investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics (project duration - 4 yrs.); Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for a study of the measurement of "total hairiness" of cotton yarn and the determination of mechanical factors contributing toward its formation (project duration - 5 yrs.); The Cotton Silk and Man-Made Fibres Research Association, Shirley Institute, Didsbury, Manchester, England, for a study of the effect of swelling agents on the fine structure of cotton (project duration - 5 yrs.), and for an investigation of chemical modifications of cotton fabrics involving control of lateral molecular order and distribution of crosslinks (project duration - 3 yrs.); Shri Ram Institute for Industrial Research, Delhi, India, for a fundamental investigation of moisture sorption and desorption by variously crosslinked cotton celluloses over the entire humidity range (project duration - 5 yrs.); State University of Ghent, Ghent, Belgium, for a fundamental study of the nature and origin of reversals in cotton fibers and their relation to mechanical properties of the fibers (project duration - 4 yrs.); Swiss Federal Institute of Technology, Zurich, Switzerland, for a study of the chemistry and structural nature of the bonds formed between formaldehyde and cellulose in formaldehyde-treated cottons (project duration - 5 yrs.); German Research Institute for Textile Industry, Reutlingen-Stuttgart, West Germany, for the development of an apparatus for counting neps in cotton card web (project duration - 4 yrs.); and Lodz Polytechnic College, Lodz, Poland, for an investigation of the mathematical and theoretical aspects of the relationship between the fiber length distribution of cotton specimens before and after sample preparation (project duration - 3 yrs.).

Chemical and physical investigations to improve products are also in progress under grants of P. L. 480 funds to the following foreign institutions: Birkbeck College of University of London, London, England, for a fundamental study of the preparation and properties of phosphazene and phosphoryl chloride derivatives having potential for reaction with cotton cellulose (project duration - 4 yrs.); The Hebrew University of Jerusalem, Jerusalem, Israel, for the synthesis and determination of properties of new aziridinyl phosphorus compounds having potential for use in the treatment of cotton (project duration - 3 yrs.); Indian Central Cotton Committee, Bombay, India, for an investigation of the preparation of radioresistant and radiosensitive celluloses (project duration - 5 yrs.); Ministry of Commerce and Industry of State of Israel, Jerusalem, Israel, for a fundamental study of the oxidation of cotton and crosslinked cotton by various oxidizing agents (project duration - 3 yrs.); Chalmers University of Technology, Gothenburg, Sweden, for a basic investigation of the behavior of cotton subjected to aerodynamic forces (project duration - 3 yrs.); Shri Ram Institute for Industrial Research, Delhi, India, for a fundamental investigation of heat and mass

transfer rates in the drying and curing of resin-treated cotton textiles by countercurrent solid-gas contact systems (project duration - 5 yrs.), and for investigations of the correlation between several important physical properties of woven cotton apparel fabrics and their performance in actual service tests, to obtain information needed for the improvement of cotton textiles (project duration - 5 yrs.); Ahmedabad Textile Industry's Research Association, Navrangpura, Ahmedabad, India, for investigation of means to minimize fiber hooked ends in cotton card and drawing slivers (project duration - 4 yrs.), and for an investigation of factors affecting drafting in the direct sliver spinning system (project duration - 5 yrs.); Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for an investigation of the effect of fiber properties on drafting tenacity during spinning of cotton and the interrelationships between fiber properties, drafting tenacity, yarn properties, and end breakage, to obtain basic information related to processing properties in the utilization of cotton (project duration - 4 yrs.); Swedish Institute for Textile Research, Gothenburg, Sweden, for investigation of the mechanism of crease formation and recovery in ease-of-care treated cotton fabrics (project duration - 4 yrs.); and Bombay Textile Research Association, Bombay, India, for a study of factors affecting curling and bursting of preponderantly warp- and filling-faced cotton fabric structure during processing of cotton into end-use products (project duration - 5 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 100.3 scientific man-years. Of this number, 24.8 is devoted to chemical composition, physical properties and structure, 28.8 to chemical and physical investigations to improve products, and 46.7 to technology--process and product development. The domestic contract and grant research involves an additional 18.7 man-years, 7.7 being on chemical composition, physical properties and structure, 6.7 on chemical and physical investigations to improve products, and 4.3 on technology--process and product development. P. L. 480 research involves 27 grants, of which 15 are on chemical composition, physical properties and structure and 12 on chemical and physical investigations to improve products.

The following lines of work were terminated during the year: (1) A determination of the structural components of the cotton fiber that contribute most to tensile strength and how they can be utilized to increase tensile and recovery properties to produce cotton products having enhanced physical properties; (2) A fundamental study of the relation of crystallinity to accessibility in native and modified cotton, to obtain information on the supermolecular structure of cotton that is needed in the development of improved cotton products (P. L. 480 project); (3) A fundamental study of the role of the structural elements of the cotton fiber in response to stress in deformation and recovery, to obtain information needed in the development of improved cotton products (P. L. 480 project); (4) An investigation of tensile and torsional or bending recoveries of single fibers, yarns, and fabrics of wash-wear treated cottons under wet and dry conditions; (5) Investigation of blending methodology to establish optimum

blending procedures for maximum utilization of cottons differing widely in fiber properties; (6) The aerodynamic separation of lint cotton into individualized fibers to provide information needed for improving cotton textile processing equipment; (7) Investigation of the effects of mechanical treatments prior to, during, and following resin finishing on the ease-of-care properties of fabrics and garments; (8) Excellent smooth drying and wrinkle resistant fabrics by crosslinking cotton with highly reactive methylolamide amino acid derivatives; (9) The development of cotton knit fabric having increased bulk, warmth, and dimensional stability; and (10) The development of cotton fabrics having improved warp and filling stretch properties by a comprehensive investigation of fabric and yarn structures and processing conditions during slack mercerization and resin treatment. The first three lines of work were under Chemical Composition, Physical Properties and Structure, the fourth under Chemical and Physical Investigations to Improve Products, and the rest under Technology--Process and Product Development.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 9.0 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition, Physical Properties and Structure

1. Fundamental Investigations of Adsorption and Swelling Phenomena in Native and Modified Cottons. Further investigations of adsorption phenomena in native and modified cottons have been made using a gas-flow adsorption technique in which surface area available to nitrogen at liquid nitrogen temperature is determined. This approach offers perhaps the most refined method of estimating the effect upon cotton fiber internal structure of chemical treatments designed to change the physical properties of the fiber. Measurements on cottons crosslinked with several different types of agents showed that the least decrease in area occurred following treatment with formaldehyde by the Form W and Form D techniques. Some crosslinked cottons appeared to have differences in pore-size distribution but these data will need to be verified by measurements using improved techniques. Parallel electron microscope observations of samples are being made in an attempt to correlate visible structural features with adsorption behavior. Selection of embedding media other than the methacrylate previously employed promises to increase the utility of metal staining procedures. (S2 1-209(Rev.)).

Basic studies of fiber property changes related to alkali sorption behavior have continued. A set of standardized conditions for routine use of the alkali sorption test (ACV procedure) was established, based on an extensive investigation of test variables. It was found that the total sorption by cotton fibers was dependent on the sodium hydroxide (NaOH) percentage (0 to 25%) of the soaking solution used in the ACV test, on the preswelling treatment of the fibers, and on fiber morphology. The sorption patterns of

the two components, i.e., NaOH and water, differed as % NaOH in the soaking solution was varied. However, from 5% to 25% NaOH, the percent NaOH in the sorbed liquid increased at approximately the same rate regardless of whether or not the fibers had been preswollen. A centrifugal acceleration greater than 100 x g is essential to obtain adequate, reproducible removal of liquid. Based on density measurements (with the dentisy gradient column) of the cottons, it was concluded that the standard ACV procedure mercerizes the fiber masses. The standardized procedure was applied to two series of treated cotton fabrics (partially acetylated, and formaldehyde treated). The extent of sorption of sodium hydroxide was found to vary with the type of modification and degree of substitution of the treated fabrics. The sorption is apparently influenced by change in spatial arrangements within the fibers, and by reactivity of the substituent with alkali (as in the case of the acetylated cottons). There are indications that the ACV procedure might be useful as a rapid test for uniformity of chemical treatment. (S2 1-249).

In contract research at Polytechnic Institute of Brooklyn, methods and techniques of measuring swelling of cotton by "rapid" procedures, such as changes in density, liquid of imbibition measurements, and changes in cross sections of fibers, have been perfected. Experimental findings on degree of swelling obtained with various nonaqueous solvents, including dimethylformamide, ethylene glycol, dimethylsulfoxide, and highly fluorinated solvents, by these procedures will be correlated with data to be obtained by X-ray diffraction, polarized infrared spectrophotometric techniques, and calorimetric methods. Changes in the fine structure of cotton which are brought about by the various solvents will be followed and mechanisms of swelling will be elucidated. (S2 1-225(C)).

Substantial progress is being made in a study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber under a P. L. 480 project at the Ahmedabad Textile Industry's Research Association, Ahmedabad, India. A proposed theoretical model for the equilibrium of cellulose fiber-aqueous system has been extensively tested through a number of adsorption isotherms for dyeing cotton with two dyes. Thermodynamic quantities such as affinities, heats and entropies of dyeing have been evaluated on the basis of the theoretical model. Vapor phase adsorption studies using nitrogen as the adsorbing molecule have given preliminary indications that there is a very marked increase in the surface area when fibers are swollen with water. The work is being extended to detect differences in adsorption of formaldehyde in Form "W" and Form "D" processes, and on specific surface areas of chemically modified cottons. Basic information being obtained is expected to be of value in improving both wet and vapor phase treatments of cotton fabrics for easy care applications. (UR-A7-(20)-46).

2. Basic Studies of the Relationships of the Structural Arrangements Within Cotton Fibers to the Physical Properties of Native and Modified Cottons. Electron microscope observations of materials crosslinked with various

derivatives of ethyleneurea have provided additional information on location and extent of crosslinking in cottons cured at various temperatures and for various time intervals in the presence of metal salt catalysts. Dimethyloldihydroxyethyleneurea proved to be more effective than dihydroxyethyleneurea as a crosslinking agent; and $Zn(NO_3)_2$ was the most effective of the metal salt catalysts investigated. In experiments on encapsulation of fibers, electron micrographs of fibers from 80-square desized, scoured and bleached printcloth treated with dimethyl silicone crosslinked with benzoyl peroxide indicated that the individual fibers were encapsulated with a thin film; in slack mercerized samples there appeared to be crosslinking throughout the fiber wall as well.

A newly developed embedding method that utilizes a water soluble medium (polyvinyl alcohol) instead of the conventional methacrylate medium is now being used in electron microscopical examination of the ultrastructure of modified cottons. This method permits better interpretation of the effects on structure of reaction both prior to and subsequent to sectioning. Structural changes associated with deacetylation of acetylated cottons of both high and low degrees of substitution have been observed using the new technique. Recent studies with the electron microscope have also indicated which concentration of sodium hydroxide used for pretreatment of cotton prior to crosslinking with butadiene diepoxide to low add-ons results in the best crosslinked products. Based on other work with diepoxide-crosslinked cottons, it appears that the application of refractive index measurements to interpretation of structural modifications may prove to be a cogent tool for evaluation of chemically treated cottons. (S2 1-263).

Fundamental investigations of plastic and oriented states of cotton cellulose have been initiated as a basis for developing durable, premanently shaped wash-wear textiles. Cotton yarn treated with sodium methoxide in dimethyl sulfoxide, or swollen in 35% benzyltrimethylammonium hydroxide, showed increased affinity for direct dyes, comparable to that of mercerized cotton, yet neither treatment caused any conversion of cellulose I to cellulose II. The affinity of cotton for direct dyes appears to be a function of degree of swelling, and is not very dependent on cellulose lattice type. It has been found that cotton yarn and fabric can be given durable shape by treatment with 35-40% aqueous benzyltrimethylammonium hydroxide or certain other quaternary hydroxides at room temperature, followed by rinsing out the agents and drying. Permanent creases have been obtained in cotton fabrics by treating ironed-in creases in this manner; false twist has been set in cotton yarn to give stretch yarns. The imparted shapes are durable to laundering but can be erased by re-application of the treating agents. Strength and edge abrasion resistance of treated areas of fabrics appear to be the same as for untreated areas.

In studies of mercerization of cotton fabric at constant dimensions, a rise in flex abrasion resistance, stiffness, air permeability and conditioned wrinkle recovery of the fabrics occurred in the concentration range of 13-18% sodium hydroxide. These results confirm those previously observed

for fabric woven of tension-mercerized yarn. New information on the mechanism of cotton mercerization has been obtained through use of combinations of quaternary ammonium hydroxides and alkali metal hydroxides. Further exploration of the plastic and oriented states of native and mercerized cotton cellulose will be carried out. (S2 1-284).

Additional research on the structure and physical behaviors of cotton fibers grown under various controlled environmental conditions (see project S2 1-217(C) below) has been conducted. The diurnal growth ring pattern observed in the cell walls of some cottons after swelling with certain cellulose solvents has been found to be a function of temperature variation during growth. When the cell walls of fibers grown under controlled conditions so as to have no growth rings were ruptured by the technique of rapid expansion with methacrylate, the resulting pattern of fracture was comparable to that obtained from fibers with growth rings--an indication that the ring is not a major plane of fracture. The presence or absence of the ring structure was also shown to be a relatively unimportant factor in mechanical properties, such as strength, elongation, and elastic recovery; and in other properties, such as dye absorption, add-on of crosslinking resin, crystallinity, cellulose orientation, and cellulose density. However, the period of oscillation for a torsion pendulum made from fibers possessing rings was slightly shorter than that of fibers without rings. A water-stress environment during growth caused a decrease of about 5° in the X-ray angle but showed no effect on strength. A 60° F. low-temperature period of growth alternated with a 90° F. period caused the strength of the fibers to be appreciably lower than when alternate 75° and 90° F. periods were used. Frequency of reversals in the fibers' winding structure is related to species: the arboreum and the herbaceum have the lowest frequency (10-20 reversals per centimeter) while the barbandense and hirsutum have the highest (20-30 reversals per centimeter). There is also an indication that reversal frequency is related to temperature of growth. This basic research is leading to new concepts concerning the structure and behavior of cotton fibers. (S2 1-208(Rev.)).

The contractor (Texas Agricultural Experiment Station) has grown cottons in chambers under additional controlled conditions for studies of fiber structure differences caused by environmental and genetic factors. Changes in rhythmic patterns of light and temperature affected the plant growth, and the cell walls of the fibers showed cuprammonium swelling patterns in accord with the changes in growth conditions. Observation of fibers swollen with cuprammonium showed that the bands of cellulose deposited under conditions of night growth at 60° F. (day growth, 90° F.) were narrower than those in fibers grown at 70° F. night temperature (day growth, 90° F.), and when temperature during growth was kept constant, no rings or bands occurred. This further confirming the conclusion that cellulose deposition is a temperature dependent function. Deltapine and Rex cottons were grown with variations of temperature, light, and water stress to alter ring structure and other properties. These varieties were also grown under constant temperatures of 75° F. and 90° F. to secure fibers without growth

rings in the cell walls but possibly having different cellulose characteristics because of the different temperatures of growth. Under the low temperature environment, the cell wall deposition was much slower than for the 90° F. environment. Measurements of various properties of these fibers are in progress. Deltapine cottons were grown under two other conditions--90° F. and 80° F., with light kept constant during the entire growth period after flowering. Fibers from bolls harvested at various intervals after flowering will be studied in detail by the Plant Fibers Pioneering Research Laboratory. (S2 1-217(C)).

Contract research at Stanford Research Institute to investigate fiber components that contribute most to the strength properties of cotton has been successfully completed. Strength improvement in cotton fibers is obtained by a combination of tensioning and swelling to cause better alignment of the structural elements of the fiber. The ratio of fiber strength at 0 gage length to that at 1/8" gage length indicates that strength improvements are a result of the reduction of weak spots along the fiber. Swelling treatments that cause the most extensive disruption of bonding forces without dissolving the cellulose are the more effective treatments. The addition of potassium thiocyanate to mercerizing strength sodium hydroxide solutions causes increased swelling, and strength increases can be accomplished essentially without changing the crystalline form of the cotton cellulose. This indicates that the more accessible regions of the cellulose are also the regions of reduced strength properties. The basic information will be helpful in developing treatments for improving the strength of cotton yarns. (S2 1-206(C)).

Research under a P. L. 480 project at the Fiber Research Institute, T.N.O., in Holland, to investigate the fundamental mechanisms and bonding forces that could be used to improve the tensile strength and other physical properties of cotton textiles is well underway. An excellent survey of current literature in this area of research has been made. Work is now progressing to determine the physical and mechanical properties of combed yarns after treatment with various media that strongly swell cotton fibers. Information growing out of research under this project is expected to be useful in improving processing treatments to yield cotton fabrics having improved strength characteristics. (UR-E19-(20)-12).

Fundamental studies of the role played by the structural elements of the cotton fiber in response to stress were conducted at the Central Laboratory, T.N.O., in P. L. 480 research completed during the past year. Through the use of modern microtechniques for manipulating and observing single fibers, a better understanding was obtained of the internal movements that occur within the cotton fiber while it is being subjected to torsion and stretching. Fibers treated by resin treatments commonly used in wash-wear finishing of cotton were found to be more rigid to torsion and to begin to form cracks and break at lower torsion than untreated fibers. Observations of stress concentrations and slip phenomena in native and resin treated fibers have furnished new data that confirm current views and extend the

knowledge of cotton fiber structure and behavior. Such basic knowledge of cotton fibers eventually will be directed toward efforts to improve cotton fiber properties through cotton breeding programs and improvements in cotton processing. (UR-E19-(20)-4).

In P. L. 480 research now nearing completion at the Swedish Institute for Textile Research, reactions that cause setting in cotton fabrics and garments have been investigated. Treatment of cotton fabrics with solutions of certain inexpensive alkalis or inorganic salts which cause swelling of the cotton fibers has been shown to cause the relaxation of internal stresses in the fabrics. This treatment, which is generally known as setting, decreases surface mussiness of the fabric, and in combination with standard resin treatments results in improved wash-wear properties. It has been observed that the conditions under which deswelling of the fibers occur during treatment greatly influence the setting effect. More recently, a study has been made of the setting effect, when used alone, of inorganic salts that are employed as catalysts in resin treatment of fabrics. Setting determinations have also been conducted under conditions of time, temperature, and concentration similar to those employed in industrial finishing, such as mercerizing, kier boiling and bleaching, to obtain a better understanding of these processes from the point of view of settings. Information developed is expected to assist in providing the basis for reducing the amount of resin required to provide acceptable wash-wear qualities in cotton textiles. (UR-E26-(20)-2).

A fundamental study of the nature and origin of reversals in cotton fibers and their relationship to mechanical properties of these fibers is underway in a P. L. 480 project at the University of Ghent, in Belgium. Examination of a number of U.S. cottons of several varieties grown in different locations has indicated that a definite, though small, effect on distances between reversals is due to growth location, while a much larger effect is due to variety. Certain botanical archetype cottons have been examined which exhibit no reversals at all, an important genetic observation. Measurement of mechanical properties of the fibers is being initiated. Fundamental information of the type being obtained in this project will be useful in relating the structure of cotton fibers to their usefulness in cotton products and will assist plant breeders in developing better cottons through the use of genetic information being obtained. (UR-E4-(20)-1).

3. Elucidation of Mechanisms of Physical Damage to Cotton Due to Mechanical, Chemical, Physical or Biological Actions. The contractor (University of Tennessee) has made good progress in the research to determine the effects of fiber extensibility on fiber breakage in mechanical processing. Techniques and instruments for impact strength testing and crushing of fibers have been developed and applied to a series of cottons covering a range in physical properties, both before and after the cottons were subjected to such treatments as mercerization, and heating at various temperatures and moisture conditions. Impact strengths for all samples except a slack mercerized cotton were found to be lower than strengths as determined by the conventional test procedure; the slack mercerized sample gave a higher

value. The changes in fiber properties resulting from crushing varied with the level of crushing--the greatest changes occurred between no crushing and the smallest pressure applied (17,000 psi). The humidity of crushing affected the change, the greatest damage being at the low humidities. The damage in mechanical working of the fibers (passing them through a granular card up to a maximum of six times) varied with cottons, pretreatments, and the humidity at the time of the working. Mechanical working shortened the fibers, increased the alkali swelling, but caused only small changes in strength and elongation. The relationships established are of importance in the mechanical processing of cotton into textile products. (S2 1-221(C)).

An investigation of the photochemical breakdown of cotton under different conditions of exposure to radiation is now reaching its final phases in P. L. 480 research at the University of Bombay, in India. It is well known that cotton fabrics are weakened by prolonged exposure to sunlight or to strong illumination. The mechanisms by which photosensitization and photolytic degradation of cotton and selected modified cottons take place have been elucidated. Photodegradation has been shown to proceed by random chain scission, the course of which is represented by two stages, each obeying first order kinetics, the initial stage proceeding at a faster rate of reaction than during the subsequent stage. The role of moisture in photochemical degradation of cotton and modified cottons has been clarified. Basic knowledge of this type is expected to be useful in devising practical means to prevent the deterioration of exposed cotton fabrics by means of chemical inhibitors or screening agents that prevent or interfere with the sequence of reactions involved. (UR-A7-(20)-4).

In P. L. 480 grant research now nearing termination at the University of Bombay, a study has been made of new, more stable solvent systems for cellulose in the determination of the average molecular weight of cellulose by the disperse viscosity technique. Copper complex solvent systems widely used for this purpose are highly colored and are extremely oxygen-sensitive, factors which greatly complicate their preparation, storage, and use. Studies have been made of several iron tartrate and cadmium ethylenediamine complex solvents that are relatively colorless and insensitive to atmospheric oxygen, and data obtained with these have been related to comparable data obtained with the older copper complex systems. Means have been developed to apply a two-component solvent system to the dissolution of high degree of polymerization cottons and mercerized samples that are difficult to dissolve in the usual solvents, thus permitting satisfactory measurement of these materials. Studies have been made of the kinetics of degradation of cellulose solutions at several temperatures and attempts have been made at fractionation in iron tartrate solvents. The information obtained in this project will be useful in following the degradation of cotton by various treatments, through the application of a simpler procedure for intrinsic viscosity measurement. (UR-A7-(20)-30).

Work is underway at the Technological Laboratory of the Indian Central Cotton Committee, Bombay, India, under a P. L. 480 grant to investigate the

microbial destruction of cotton fibers and fabrics that occurs in exposure during outdoor uses. A number of bacterial and fungal cultures have been isolated from cotton fabrics exposed to outdoor environments in the Bombay area. A study will be made of the mechanisms by which these organisms cause rotting or tendering of cotton fabrics. Basic knowledge of this type will be useful in devising improved treatments to prevent or minimize biological degradation of cotton fabrics in applications such as tentage, tarpaulins, awnings and boat covers, which represent substantial markets for cotton. Protection of these markets from inroads by synthetic fibers through the development of highly effective, more permanent biocidal treatments for cotton fabrics is urgently needed. (UR-A7-(20)-32).

4. Investigation of the Structural and Compositional Changes Occurring During Chemical and Physical Modification of Cotton Cellulose. Pioneering research on plant fibers has been continued along a number of lines by the Plant Fibers Pioneering Research Laboratory. The objective of this Laboratory is to obtain basic information on the fine or supermolecular structure of plant fibers; and to relate the information of polymer and fiber structure to the mechanical and textile behavior of fibers.

During the past year the mechanism of the heterogeneous reactions of cellulose with many chemical substances--acetyl, propionyl, and benzyl compounds, and acrylonitrile--has been very much clarified. It has long been recognized that cellulose is organized in nature as a highly crystalline material; and it also is generally recognized that chemical agents are usually excluded from reaction in the crystalline regions although many agents (for example, strong alkali) are able to penetrate by chemical means and disrupt the crystalline organization. As a result of recently completed studies it is concluded that in many heterogeneous cellulose reactions two diffusion-controlled processes proceed simultaneously. One of these, "decrystallization," has the effect of supplying new, readily accessible cellulose for the reaction at the expense of the crystalline fraction. The other, a chemical reaction process, depends on the rate of transport of the active agent by diffusion to the site of reaction. The consequence of the two processes is to yield a two-stage rate curve. During the first stage, when both crystalline and amorphous cellulose are present, reaction takes place both in the originally accessible cellulose and in the cellulose newly made accessible. Therefore, the rate of reaction is highest. During the last stage, when the crystalline cellulose has become exhausted and only the amorphous cellulose is present, only the residual accessible cellulose undergoes reaction and the rate is noticeably decreased. Previously, it was supposed that the rate should increase rather than decrease when only the amorphous component remained. However, this view failed to take into account the continually expanded supply of amorphous cellulose present during the first stage of the reaction, and the diminished supply remaining during the last stage of the reaction. Apparently, the newly recognized mechanism will be applicable in all those cases where the reagent can disrupt the crystalline structure. The extent of its applicability is being studied. Meantime, the results have been summarized for publication.

Another line of work has been concerned with crystalline structure and orientation of single cotton fibers. The general excellence and versatility of cotton as a textile material largely arises from the spiralling fibrillate texture of the cellulose which makes up the bulk of the fiber. The mechanical properties of the cotton have long been known to be correlated with the crystalline nature of the cellulose and the orientation of the molecular chains with respect to the fiber axis. But the exact nature of the correlation has not been clear. A detailed examination of these aspects of the fiber structure was undertaken by X-ray diffraction measurements on single fibers, and extended to optical orientation measurements to develop corroborative information.

Diffraction patterns of 100-micron lengths of single fibers were obtained with a microcamera. Subsequent photometering of these patterns showed that the (002) interference, which appears as a single arc in macrodiffraction (fiber bundle) photographs, was resolved azimuthally into two spots. These spots arise from the opposite orientations of the crystallites in the front and back walls, respectively, of the fiber. The resolution of the (002) arc in these experiments appears to be the first reported. Thus, a direct measurement of the mean angle of spiral orientation of the cellulose crystallites in cotton has been accomplished.

The orientation angle of the cellulose in the wall of the fiber with respect to the fiber axis was also measured by polarized light microscopy, using fibers which had been axially fractured after weakening and embrittling by crosslinking, hydrolytic, or oxidative pretreatments. The results of these measurements were in good agreement with those of the single-fiber X-ray measurements, and the lengthy X-ray exposure time required may thus be avoided.

On the assumption that the bundle diffraction pattern consists of numerous individual well-resolved single fiber patterns which have been superimposed on each other, a mathematical expression relating the fiber spiral angle to the diffraction intensity distribution of the (002) arc of the bundle pattern has been derived. From this relationship, the widely practiced measurements of the "50%" or "40%" X-ray angle (angle between maximum and 50% or 40% of maximum intensity, respectively) may be used to derive the mean spiral angle of orientation of the cotton. The mean spiral angle of the fiber is then simply the "75%" X-ray angle.

The conclusions drawn from this research are that the mean spiral angles of the fibers of all cottons are not the same, but range from approximately 18° to 25°; and that the spiral angle reaches a limiting value as the 50% X-ray angle becomes larger than about 30°.

Crystalline characteristics of cellulose and cellulose derivatives have also been investigated. An adequate description of the structure of cellulosic fibers must place considerable emphasis on their crystal character and its response to various physical and chemical treatments. X-ray diffraction

continues to be the chief tool by which crystallite size, degree of crystallinity and crystal geometry are measured. These parameters, along with crystallite orientation, give basic information needed to determine the supramolecular arrangement of any fibrous polymeric material.

The Scherrer line-broadening technique was applied to the (002) and (040) crystal lattice interferences, which give measurements of the diameter and length of the crystallites, respectively. The crystallite diameters of cottons from several species varied ober a relatively narrow range (50-60 Angstrom). Ramie fibers showed a value in this same range, while Chinese wool cotton showed a slightly lower (45 Angstrom) diameter. Correction of the data for amorphous content of the cotton did not appreciably affect the values obtained. Examination of chemically modified cottons (e.g., esters) indicated that crystallite diameter is diminished.

Crystallite length in cottons and ramie has been found to range from 200 to 240 Angstrom units. This value is far short of the previously accepted minimum value of 600 Angstrom units. The higher precision of the counting technique used in the present studies as compared to the photographic measurements of earlier workers lends a high degree of confidence to the present results. It was found that the crystallite length of propionated cotton cellulose increases significantly as the degree of substitution (D.S.) progresses to a level of 2.0. This observation is surprising, since the crystallite diameter is concomitantly decreasing. However, it may be explained in terms of the swelling and tension existing in the fibers during esterification.

An important application of the crystallite size data being obtained is in the determination of the true arrangement of the cellulose chains in the microfibrillar units of the fiber. The possibility of cellulose chain folding, which is being strongly advanced by a number of workers in the field, can probably be verified when sufficient data on crystallite dimensions of variously treated celluloses has been made available.

The measurement of the degree of crystallinity of cottons by X-ray methods requires that the crystalline standard used be of the same crystal modification as the sample. Determination of the degree of crystallinity by the Wakelin method, using a computer-programmed technique, has shown that the crystallinity of commercially mercerized cotton yarn is approximately 65%, and this value is increased to 70% by further treatment with mercerizing strength sodium hydroxide.

Study of the changes in the crystal lattice of cellulose on esterification or etherification has shown that new interplanar spacings appear in every case at the expense of the original (101) spacing. The application of these observations is that in the reactions the C_6 hydroxyl is the primary site of chemical attack. The distention of the original lattice is, in general, proportional to the size of the substituent group introduced, when the group is essentially linear. However, for bulky groups such as the

methyl ester of camphoric acid, the increase in interplanar spacing is less than would be indicated by the extended length of the group. It may be concluded that certain bulky substituents are able to take up favorable positions for minimum spacing between adjacent cellulose chains.

Investigations of the effect of carefully controlled environmental growth conditions on the fine structure of cotton fibers at different stages of their development have been continued and definite progress has been made. Five series of bolls grown in environmental chambers under specified conditions were provided by Texas Agricultural Experiment Station (contract project, S2 1-217(C)) during the year, making a total of six. Two more series are being grown. All series were grown under continuous illumination throughout the growth period of the fibers; the temperatures were either held constant (at 90°, 80°, or 70° F.), or were alternated on a 15-9 hr. cycle (90°-70°, or 80°-70°F.). Immature bolls harvested at 5-day intervals, beginning at 20 days after blossoming, are preserved in two ways: (1) by freeze-drying, and (2) in methanol. Mature bolls harvested after opening on the plant are also being studied.

Analyses for moisture, sugars and cellulose content (completed on three series) give criteria of relative rate of development under different conditions. So far (two series) increases in crystallinity during growth, as indicated by X-ray diffraction, have not been found to be large. For example, the X-ray crystallinity of the youngest fibers was about 77-78%, that of the mature fibers about 87%. Density data are in accord with these findings; the density increased slightly as the fibers matured.

Accessibility, measured both by dye sorption and by enzyme attack, decreased considerably as the fiber matured. In this connection, a simple method of using a cellulolytic enzyme for quantitative measurement of accessibility has been developed. A colorimetric microdetermination of cellulose, based on a colorimetric sugar test, has also been developed for measuring the cellulosic residue after enzyme attack.

Light- and electron-microscopy of selected samples is beginning to yield some interesting information about fine structure. However, observations are still too limited to warrant definite conclusions.

In continuation of a study to characterize and more fully interpret the nature of the microfibrillar organization of cellulose, samples of cellulose I, II, III, and IV were prepared for use in molecular-weight distribution studies. By means of viscosity measurements in cupriethylenediamine hydroxide solution (cuen), the degree of polymerization (DP) for each of the polymorphic forms was established. DP values of 4300, 3530, 3210, and 1670, respectively, for the celluloses I, II, III, and IV were obtained. Portions of the samples were hydrolyzed in boiling 2.5 N hydrochloric acid for 1/4, 1/2, and 1-hour to provide leveling-off DP hydrocelluloses. These materials will be studied closely with respect to new views regarding chain folding in cellulose.

In preparation for column fractionation, nitration of the celluloses was carried out to the trinitrate stage. Viscosity measurements on the nitrates in ethyl acetate solution paralleled those made in cupriethylenediamine. Cadoxen viscosity determinations were also made. With this solvent the DP's of the samples were found to be 5030, 4460, 4930, and 1860, respectively, for cellulose I, II, III, and IV. These values are higher than those obtained with the cuen solvent, due, it is believed, to less oxidative depolymerization.

Distributions of molecular weight were obtained on the trinitrates of the four celluloses by gel permeation chromatography. Weight-average DP's ($\overline{DP}_w$) were 20, 940, 18,200, 18,930, and 13,180, respectively, for celluloses I, II, III, and IV; or number-average DP's ($\overline{DP}_n$), 12,570, 9,050, 9,780, and 6,680, respectively. These high values, relative to the cuen and cadoxen values, are believed to be realistic and in line with reports in the literature giving values differing by like magnitude for different methods of determination. Polymolecularity of the cellulose, as indicated by the ratio $\overline{DP}_w/\overline{DP}_n$, was normal, being 1.67 for the cellulose I and 1.9 - 2.0 for the others. Shifts in both the differential and integral distribution curves, however, indicated that the processes leading to the lattice conversions had caused significant changes in the molecular chain-length distributions.

A rather comprehensive laboratory study of the preparation and mechanical and thermal properties of propionylated cotton has been initiated. Some preliminary results of this research are reported here. Although cellulose propionate is well known in the literature, the introduction of the propionyl group into the cellulose of cotton fibers without loss of the fibrous structure has scarcely been considered. All degrees of substitution from minimal to essentially complete are evidently possible, and these can have different and important effects on the structure and the chemical and physical properties of textile products prepared from the modified cottons.

A series of uniformly substituted propionylated cotton yarns (D.S. = 0.24 to 2.94) were made by reacting 7/2 mercerized yarn with propionyl chloride in pyridine and dimethyl formamide for different times. The reaction conditions, concentration of the reagent and effect of tempreature, were studied. For complete substitution it was necessary to carry out the reaction in two steps in which the sample was washed, dried, and rewrapped inside out and then reacted again. It was found that D.S. computed by weight increase far exceeded that by saponification (although the relationship was linear) and in the completely reacted sample was much higher than the theoretical value of 3. Gas-liquid chromatograms also showed an additional peak which could not be accounted for by the expected decomposition products. Several types of experimental evidence were obtained which support the idea that a side reaction forming propionyl propionate occurs simultaneously during the reaction. Such a reaction has also been postulated by Malm and coworkers but they did not mention the reasons behind their postulate.

It was found that the Sakurada plot of the D.S. vs reaction time of the

propionylated samples gives two intersecting straight lines. This supports the idea of Conrad and Chatterjee that two reaction rates, decrystallization and reaction, are involved. These work additively in the initial stages until the crystalline portion is exhausted and then only the latter continues in the residual accessible cellulose. The slope of the straight line at the latter stage probably gives the exponent of the rate equation in the amorphous phase. Further evaluations were made by the measurement of density, differential thermal and thermographic analysis (DTA and TGA), X-ray diffraction and measurement of tensile properties with an Instron Tester with and without rise of temperature, with the following broad preliminary conclusions: (a) There is a linear relationship between density and percent propionyl content; and between specific volume and D.C. (b) Exothermicity of the DTA curves decreases with substitution, consistent with its lower cellulose content. (c) DTA and TGA tracings indicate lower crystallinity with substitution. (d) Mechanical testing with the Instron Tester shows that yarn linear density (Tex) increases with D.S. and that tensile strength remains within 90% of the control up to a D.S. = 1.3 and then decreases. Elongation reaches a maximum at 1.3 D.S.; tensile stiffness decreases and toughness increases with D.S. It was found that log stiffness vs D.S. and fiber density vs stiffness follow linear relationships.

In a continuing study of the relationship between the configuration of an acid chloride and its reaction with cellulose, the reactivity of the optically active and racemic methyl camphoryl chlorides toward cellulose was investigated. Because of conflicting information in the literature, work with the monomethyl camphorates was necessary to clarify the structures of the acid chlorides which were to be reacted with the cellulose. It was found that thionyl chloride, by an as yet unknown mechanism, converts the α-methyl camphorate into the acid chloride of β-methyl camphorate. This acid chloride then reacts with cellulose to yield α-cellulose β-methyl camphorate. Thus, whether one starts with the α-methyl camphorate or the β-methyl camphorate one obtains the same cellulose derivative. In this derivative, the ester linkage to cellulose is somewhat hindered, but distinctly less hindered than the ester linkage to the methyl group. For this reason, upon saponification, one obtains cellulose and not the half ester α-cellulose camphorate.

Samples of d-, l-, and dl-β-methyl camphoryl chloride were prepared and reacted with cellulose in pyridine under similar conditions. The tentative conclusion is that the optically active and the racemic forms react with cellulose at essentially the same rate; however, further experiments are in progress.

All of the cellulose methyl camphorates are white in color and retain their textile form. When the densities of the cellulose derivatives made from the d-, l-, and dl-β-methyl camphoryl chlorides were plotted versus the degree of substitution (D.S.), a single smooth curve was obtained.

Considerable information was also obtained on the methyl isocamphorates. Thionyl chloride does not convert α-methyl isocamphorate into the β-methyl

isocamphoryl chloride. This finding will be of considerable value if the preparation of cellulose methyl isocamphorates is undertaken. (SU-P-2).

Several related lines of research--not conducted in the PF Pioneering Research Laboratory--are also in progress. One of these involves a spectroscopic investigation of the molecular changes in structure occurring during the chemical modification of cotton cellulose. Techniques were recently developed for obtaining suitable spectra of cellulose and its derivatives in the near and far infrared regions. Increased knowledge of the molecular structure of cotton and modified cotton may be gained by application of these techniques. Thus far, spectra of cellulose I, II, III, III_2, and IV and of certain sugars have been obtained by the potassium bromide disc method in the regions from 0.7-3μ and 800-300 cm^{-1}. Tentative band assignments for these regions of the cellulose spectrum will be made. Preliminary experiments indicate that infrared multiple internal reflectance spectra may prove useful in following changes in fabrics subjected to weathering. (S2 1-287).

Basic investigations of accessibility to complexing agents of stable cotton cellulose derivatives have been initiated. By means of X-ray diffraction analysis, decrease in crystallinity and change in crystalline lattice type have been examined for a series of cotton fabrics crosslinked with formaldehyde to various levels by the Form W and Form D processes and treated with selected swelling agents. Information on the changes in accessibility as functions of amount of crosslinking agent was developed. As the amount of crosslinking increased, there was a decrease in conversion of cellulose I to cellulose II for a given fabric. Even at the highest level of formaldehyde, the crosslinks had not made the cotton completely resistant to attack by swelling agent. The degree of restriction varied with the particular agent. Knowledge of the structural details of modified cotton products will be of value in furthering basic and applied developments in this research area. (S2 1-280).

A promising new X-ray diffraction technique for determining the swelling ability of liquids in contact with cotton cellulose has been investigated. Preliminary results lead to the belief that it will eventually provide a quantitative technique for the evaluation of fine structure changes in cotton in contact with liquids. Further evidence of the effect of moisture in increasing the crystallinity of cellulose over that of the dry condition has been obtained, using cotton partially decrystallized by vibration ball milling, and Avirin, a commercially obtained hydrocellulose. Butylamine has been found to swell cotton directly at room temperature, producing the butylamine-cellulose complex. At a temperature of 6°C. it is even more effective, giving essentially complete transformation to the complex. The effectiveness of a number of commonly used swelling agents will be evaluated using the new X-ray technique, and the results will be compared to those obtained using other techniques for evaluation of swelling in cotton cellulose. (S2 1-276).

Studies of the fluorescence of native and modified cottons have continued. Satisfactory spectra have been obtained from a number of native and modified

cottons, in both yarn and fabric form. Excitation maxima ranged from 300 to 420 millimicrons, and emission maxima ranged from 350 to 500 millimicrons. Thus, chemically modified cottons show considerable variation in their fluorescence spectra. Emission and excitation spectra obtained on samples of modified cotton celluloses in potassium bromide discs were generally weak; it is anticipated that this technique will be of value only in those instances when a small sample is available. It was found that extracts obtained by extraction of grey cloth with a number of different solvents usually exhibited fluorescence, which indicates that the noncellulosic constituents of cotton play a role in the interaction of light and cotton. This may prove of value in studies of cotton degradation in sunlight. Instrumentation for the observation of light emitted by cotton at elevated temperatures (oxyluminescence) has been completed and is undergoing checks. Preliminary results indicate that the oxyluminescence can be used to calculate activation energies and therefore may be of value in the study of the oxidative degradation of cotton products. In the course of analyzing and correlating data from fluorescence excitation and emission spectra, infrared FMIR spectra, and reflectance spectra of a number of modified cottons on which oxyluminescence measurements are available, it was observed that the fluorescence spectra of the control (unheated) cottons did not agree with previous spectra of the same cottons. An explanation for this is being sought. (S2 1-264).

New basic information concerning the reactions of cellulose and related model compounds is being obtained by use of nuclear magnetic resonance (NMR) spectroscopy. Efforts to react dimethylolethyleneurea (DMEU) with compounds structurally related to cellulose have met with limited success. However, the results thus far obtained seem to indicate that DMEU may be reacting with the hydroxyl of the model compounds through only one of the alcohol groups. The assignment of peaks in the NMR spectrum of the acid hydrolyzate of DMEU has been made. This information will be useful in the research on reactions of this crosslinking agent with the model compounds. Correlation of such reactions with the corresponding cellulose reactions may aid in the elucidation of the chemical structure of modified cottons. Other work has shown that the acid hydrolyzate of dimethylol ethyl carbamate (DMEC) or of DMEC-treated cotton fabric contains the monomethylol derivative as one of its components. (S2 1-268).

Basic and exploratory studies have been initiated to relate the magnetic, vibrational, and electronic spectral properties of cotton finishing chemicals to their suitability for imparting valuable properties to cotton textiles. Data from nuclear magnetic resonance spectra of selected compounds used as finishing agents are being collected and analyzed. Where possible, complete series of compounds of several important types will be secured and their spectral properties determined. (S2 1-291).

Recent research on separation and identification of cleavage products of partially etherified cottons has provided information on the sites of reaction in the anhydroglucose unit and the relative distribution of substituents for cottons modified with methyl vinyl sulfone (MVS), hydroxyethyl

methyl sulfone, diethylaminoethyl (DEAE) chloride, and sodium allyl sulfate. For MVS-treated cotton, the ratio of monosubstituents at the 2-O-, 3-O-, and 6-O- positions on the anhydroglucose unit is 0.20 : 0.04 : 1.0. The mole ratio of mono- : di- : trisubstituted components in the products from a single MVS-treatment of cotton was 1.0 : 0.05 : ca. 0.0; that from a triple treatment was 1.0 : 0.23 : 0.12. Samples of cotton treated with hydroxyethyl methyl sulfone showed distribution of substituents in the 2-O-, 3-O-, and 6-O- positions of the order 0.3 : 0.1 : 1.0. The presence of the 2-O-, 3-O-, and 6-O-diethylaminoethylglucoses in the hydrolyzate of DEAE cotton of DS 0.8 has been established; the distribution occurs in the ratio 1.25 : 0.35 : 1.0. Some di- and trisubstituted products are also present. Preliminary analyses of the monoallylglucoses from allyl cotton indicate a distribution of 2-O-, 3-O-, and 6-O- substituents in the ratio of 1.2 : 0.2 : 1.0. This research is leading to a better understanding of the chemical modification and crosslinking of cotton cellulose. (S2 1-214(Rev.)).

The relative efficiencies of crosslinking and number of effective crosslinks for a variety of types of formaldehyde-modified cottons have been estimated from sol-gel and distention index (i.e., apparent specific volume) measurements. The data represent the first quantitative information of this type on crosslinked cottons. It was established that the degree and type of heterogeneity of distribution of the formaldehyde crosslinks is a function of the process of reaction, depending to a large extent upon the relative rates of the chemical reaction and of the diffusion of reagents. Each specific process for introduction of the formaldehyde crosslinking agent is characterized by a completely unique relationship among distention index, gel fraction and reagent content. Efficiencies range from as low as 0.007 for Form D' cotton to as high as > 0.15 for Form W' cotton (relative to the ideal of model system as 1.0). The number of effective chain segments between crosslinks estimated at 0.20% level of formaldehyde in these cottons is $<< 0.03 \times 10^{-5}$ for Form D' to 4.8×10^{-5} for Form W' cotton; the number of effective crosslinks is one-half these values. It is pertinent that the crosslinked cotton exhibiting the highest number of effective crosslinks (Form W') has approximately one-third the number that is ideally possible. In other work, the molecular sieve characteristics of ball-milled cotton and crosslinked ball-milled cotton (Form D), as measured by gel permeation chromatography, were found to differ, an indication of differences in their internal structures. Further use will be made of this promising new investigational tool. (S2 1-255).

In a P. L. 480 project at the Swiss Federal Institute of Technology, Zurich, Switzerland, a study is being conducted of the chemistry and structural nature of the bonds formed between formaldehyde and cellulose in formaldehyde-treated cottons. Means have been developed for introducing high levels of formaldehyde into cotton, and improved analytical methods for determination of formaldehyde contents have been devised. Work is now moving into the area of structural studies on samples of cotton crosslinked with formaldehyde by different processes. Since the formaldehyde crosslink is widely used and in treating cotton fabrics to confer wash-wear properties,

the basic data obtained in this project is expected to provide information that can be translated into practical use in devising improved cotton products. (UR-E27-(20)-2).

A fundamental investigation of the effect of swelling and stretching treatments on the fine structure and mechanical properties of cotton fibers is now moving into its final phase under a P. L. 480 grant at the Ahmedabad Textile Industry's Research Association (ATIRA), in India. The effect on fiber fine structure, as revealed by X-ray, microscopic and modulus measurements, of swelling fibers under tension with agents such as solutions of sodium hydroxide, ethylene diamine, and zinc chloride has been studied. An instrumental technique for measuring the viscoelastic properties of modified cottons has been developed. It has been shown that orientation is considerably more decisive than crystallinity in determining the elastic modulus of cotton fibers. The information obtained in the investigation is expected to be useful in the selection of treatments to improve the mechanical behavior of cotton products. (UR-A7-(20)-19).

In a P. L. 480 project completed during the past year, a basic study of the fine structure of the cotton fiber was conducted at the National Institute of Applied Chemical Research in Paris, France, to relate fine structure to other fiber properties that are important in the processing and use of cotton. Refined physical and chemical techniques, including X-ray diffraction and microcalorimetry were applied to the measurement of fine structural features and behaviors of a typical U.S. cotton of Deltapine variety for which extensive fiber data were available, and to the same cotton treated at several levels of three well-known crosslinking treatments. Extensive data on parameters such as moisture sorption, swelling, crystallinity, and specific surface area were collected and subjected to analysis and interpretation. Moisture sorption was affected differently by the several crosslinking treatments but all of these increased the mercerization threshold of the cottons. Crosslinkage by formaldehyde lowered crystallinity as compared with the control native cotton. The information obtained eventually will be translated into the development of improved cotton products. (UR-E9-(20)-61).

A P. L. 480 project is underway at the Shirley Institute, Manchester, England, in which a study will be made of the effect of caustic soda and other swelling agents on the fine structure of cotton. The first phase of the project, in which a comprehensive and critical survey of the literature on the swelling of cotton was made, is now completed. The manuscript is now in the hands of the printer and copies are expected to be ready for distribution in the near future. It is felt that this survey will fill an urgent need of researchers in the field of swelling and in the practical implications of swelling in cotton processing. Work is now well underway in the second phase of the project, which is designed to fill gaps in the literature of swelling made apparent in the survey. (UR-E29-(20)-65).

5. Relationship of Gross Structure of Cotton to Behavior of the Fibers in

Textile Structures. Substantial progress has been made in basic research under a grant at Georgia Tech Research Institute to elucidate the role of fiber morphology in frictional behavior. The research is seeking to delineate the respective effects of fiber shape, surface texture and internal structure on the friction between contingent fibers; to evaluate the relative influences of discrete fiber properties on friction of fibers in aggregates; and to relate these properties to the behavior of fibers in various processing stages. An electromagnetic, servo-controlled fiber friction apparatus for measuring friction of single fibers has been developed but certain refinements will be needed to improve its discrimination, accuracy, and reliability. With this apparatus, the coefficients of friction for cotton fibers of different length (3/4, 1 and 1-1/4 in.) from a single sample were found to have essentially the same values, .39, .44, and .44, respectively. The slipstick behavior differed for cotton, dewaxed cotton and nylon. For nylon the slips were more frequent (about 7 per mm.) compared with natural cotton (between 3 and 4 per mm.); the number for the dewaxed cotton was intermediate. When a slip occurred in the dewaxed cotton the excursion frequency was very large. In another phase of the work, crimp and convolutions have been characterized for two cottons. Electron photomicrographs of the surfaces characterized for friction will be made, and relationships between fiber surface and friction will be established (S2 1-248(Gr.)).

Mathematical analysis of the basic mechanical and geometric factors which influence the bending of a yarn structure has been continued in grant research at the Massachusetts Institute of Technology. Particular emphasis has been given to the role of interfiber friction as it affects the movement of fibers within a yarn, and its effect on bending recovery as contrasted to permanent deformation of the material. A generalized theory of the behavior of multiple-layered beams under deformation (with friction) has been derived. A newly designed test instrument to measure the moment-curvature relations in bent yarns has been completed, and the experimental results obtained with it have been compared with those predicted from the theory. From results thus far obtained, it is clear that friction plays a significant part in the bending and recovery of single yarns. This research is expanding our knowledge of the configurational changes that take place in cotton textile structures during bending and wrinkling. (S2 1-237(Gr.)).

Research is progressing under a P. L.480 grant at the Juan de la Cierva School of Technical Investigations, Barcelona, Spain, to devise means for the measurement of "total hairiness" of cotton yarns and to determine mechanical factors that contribute to the formation of this phenomenon in the spinning of cotton. It is thought that higher spinning speeds cause increased "hairiness," a fuzzy condition resulting from the protrusion of fiber ends from the body of the yarn. Since for many uses yarns are singed to remove this hairiness, the amount of fibers so removed could have important economic implications. An improved transistorized prototype electronic apparatus to measure and record the "total hairiness" of cotton yarns is in the final stages of completion. A second phase of the project,

in which the apparatus will be employed to evaluate factors in spinning that contribute to yarn "hairiness," will soon get underway. Delineation of the factors causing yarn hairiness is expected to be useful in that it will permit machine adjustments to be made in processing cotton to minimize the formation of hairiness in yarns. (UR-E25-(20)-31).

6. Investigation of Factors Influencing Comfort in Cotton Textiles. The contractor (Harris Research Laboratories, Inc.) has made good progress in research investigations of factors influencing comfort in cotton apparel fabrics. The sources of decreased comfort in resin-treated wearing apparel are being progressively identified. The changes in surface character of the fabric as moisture from perspiration is built up under warm stress appears to be the dominant phenomenon. A consistent picture is emerging, of groups of subjective comfort sensations which can be definitely associated with fabric surface variations due to the presence of resin, the concentration of resin, and fabric structure. Fabric stiffness seems to be an important property in eliciting differences in comfort response. The use of softer fabrics for shirting materials would appear to permit a higher degree of comfort. Means for changing the moisture distribution in fabric surface regions also appears desirable, since differences in distribution of moisture in the fabric surface have been found to be more important than moixture content alone. A device for quantitatively evaluating this moisture distribution, which has been demonstrated to be a key factor in comfort sensations, has been constructed and is being evaluated. (S2 1-241(C)).

7. Development of New and Improved Methods and Instruments for Measuring the Physical Properties of Cotton and Its Products. In research to develop more reliable methods of appraising abrasive damage on all-cotton wash-wear fabrics, two simplified test procedures have been devised for rapid determination of edge abrasive damage on delayed cure type fabrics. One of these procedures, a simplified Accelerotor edge abrasion test, has shown very good reproducibility, and its evaluation is continuing. Results obtained with photographic standards devised for rating Accelerotor edge abrasion of crosslinked twill fabrics correlated highly with results obtained using the actual fabric standards. Use of photographic standards will facilitate the standardization and comparison of interlaboratory test results. The second test procedure under evaluation is the Launder-Ometer edge abrasion test; results obtained with it haven't been as encouraging. Other preliminary tests have indicated that a commercial, fugitive softening agent, as used by the housewife in home laundering, increases the flex abrasion resistance of desized, scoured cotton fabrics. (S2 1-275).

The final design for a prototype instrument for counting neps has been completed by the contractor (Stanford Research Institute). The instrument consists of a deflocculation system that presents the fiber, neps and foreign material to the air stream, followed by two stages of nep purification, one stage consisting of aerodynamic trash separation and the other of electro-aerodynamic nep separation. It is doubtful that 100% nep purification will

be possible and therefore the instrument has been designed to incorporate polarizing filters in the optical system which will enable discriminating against stray fibers and flock that might carry over into the discriminator. Means for applying an electrostatic field to align single fibers is being provided as a further aid to discrimination between neps and fibers. Fabrication and assembly of the prototype are in progress. The successful development of a method and instrument for counting neps in raw cotton and at the various stages of textile processing will aid considerably in improving textile processing techniques and equipment. (S2 1-229(C)).

The objective of another contract project at Stanford Research Institute is to develop a research instrument for accurately and automatically determining length, length distribution, and diameter of cotton fibers in a continuous, automated operation. Such an instrument would aid in improving the efficiency of cotton textile processing and in producing cotton products with improved properties. Systems for the evaluation of concepts for presenting fibers to light scanning units have been theoretically evaluated and equipment for experimental evaluation has been designed and fabricated. The results of preliminary measurements of fiber light scattering were judged inadequate as a basis for the design of the photometric portions of the instrument for determining fiber length and diameter distribution. Accordingly, the light-scattering specimen mounts were redesigned to facilitate a thorough investigation of light scattering by individual fibers. Construction and evaluation of the new setup is progressing. (S2 1-266(C)).

P. L. 480 research at the German Research Institute for Textile Industry to develop an apparatus for the rapid and automatic counting of neps in cotton card web by means of light reflectance and detection is now entering its final phases. Principles developed through studies using a prototype instrument that scans a sample of card web have been extended in two promising approaches toward the problem of measuring neps in the running web on the full-scale card. The approaches involve the use of some of the principles of television for scanning the web and clever, sophisticated electronic instrumentation has been devised and evaluated. Applications for patents covering the instrumentation developed have been filed in the U. S. and in West Germany. The availability of a suitable instrument to measure neps in the card web is expected to be of great value to cotton processors since it would provide rapid means for following, and perhaps automatically controlling, an important processing variable that affects cotton fabric quality. (UR-E10-(20)-2).

An investigation of the mathematical and theoretical aspects of the relationship between the fiber-length distribution of cotton specimens before and after sample preparation is being conducted under a P. L. 480 project at the Lodz Polytechnic College, Lodz, Poland. Work is underway to establish the change in relationships between the length distributions of cotton fibers resulting from actions of mechanical processing steps. This problem is of greater importance for cotton than for synthetic fibers because the wide range of fiber lengths in cotton makes breakage during processing less

apparent than for synthetics, in which the fibers tend to be initially of the same length. Excellent progress has been made in the theoretical approaches, and refinements such as the effect of taper on each end of the cotton fibers are now being built into the mathematical model. Developments under the project will be of exclusive benefit to cotton and should permit more sophisticated analyses of the effects of various processing stages on the fiber length distribution of cottons. (UR-E21-(20)-27).

B. Chemical and Physical Investigations to Improve Products

1. Exploratory Chemical Modification and Finishing of Cotton. Research on improved methods of etherifying cotton cellulose and production of fibrous ethers, thioethers and other derivatives has continued. Pretreatment of cotton by slack mercerization and solvent exchange has led to an improved rate of reaction with thionyl chloride in the preparation of chlorocellulose, a useful intermediate for producing ethers and thioethers. Chlorocellulose yarn has been used to produce a phenylthiocellulose product and a 1,4-butanedithiol reaction product. Cuene-insoluble products with 21 to 46% add-ons have been obtained by treatment of mercerized cotton yarn with dichlorosulfolane. Use of buffer with disodium methylene sulfate in pad-cure treatment of fabric has been found to result in improved strength retention; however, wrinkle recoveries of the fabric are lowered somewhat. The benzylation of cotton yarn at room temperature to a D.S. of 1.5 has been accomplished using sodium methoxide in dimethyl sulfoxide as the activator. Phosphorylated cotton obtained by the rapid and inexpensive process of curing fabric with sodium acid phosphate is proving to be a useful reactive material for further chemical modification. The rate of graft polymerization of acrylonitrile (or this monomer mixed with hydroxypropyl methacrylate) on phosphorylated cotton fabric is much greater than on unmodified cotton. The phosphorylated cotton is also reactive towards mercaptans, giving a new type of cellulose derivative. Emphasis in future work will be on cellulose derivatives prepared from chlorocellulose and on the evaluation of various graft polymers on phosphorylated cotton. (S2 1-219; S2 1-283).

In research supported by the Cotton Producers Institute, three chemical reactions which exhibit reversibility potentially suitable for reversible crosslinking and creasing of cotton have been found and are under investigation. These are: carbamate dissociation, Diels-Alder dissociation, and malonic ester transesterification. Results of preliminary evaluations of ethyl N-(p-glycidoxyphenyl)-carbamate and bis-N,N'-(carbophenoxy)-p-phenylenediamine as thermally reversible crosslinking agents for cotton fabric were not too encouraging. Isomers and esters of dicyclopentadiene-dicarboxylic acid (DCPDA), a compound that undergoes a reverse Diels-Alder reaction at elevated temperatures, are being investigated. Kinetic studies of thermal dissociation of the methyl ester of DCPDA in homogenous solutions indicated that this dissociation reaction occurs at a rate adequate for a reversible crosslink in cotton. Thermally reversible crosslinks were successfully introduced into cotton printcloth by esterification with DCPDA, as evidenced by the fabric's increased wrinkle recovery and by the

introduction of creases when the esterified fabric is held in a creased configuration at elevated temperatures for periods approaching three hours. Normal laundering and ironing procedures do not remove the creases but they can be removed by heating the fabric in the flat configuration for similar periods of time. The extended heating times required were not anticipated, based on results of the kinetic studies. In research on malonic ester transesterification, the observation that a facile ester interchange occurs between diethyl malonate and isoamyl alcohol at temperatures as low as 150°C. led to studies of the crosslinking of cotton fabric with malonic acid. The crosslinked fabric exhibited increased wrinkle recoveries, and durable creases could be imparted by holding the fabric in a creased configuration at 150°C. for 24 hours. The ultimate goal of this exploratory research is the development of a crosslink that is sufficiently reversible at elevated temperature for practical thermally reversible creasing in a wrinkle resistant cotton fabric. (S2 1-258).

Exploratory investigations of spatial and structural effects of reversible and conventional crosslinks in cotton have continued. Research on the thermal reversibility of ester crosslinkages in cotton via transesterification, as measured by the thermal introduction of durable creases, has shown that cellulose esters of polycarboxylic acids containing free neighboring carboxyl groups--for example, of cyclopentanetetracarboxylic acid--are more readily reversed than those of simple di- or tricarboxylic acids. In situ catalysis apparently occurs, one neighboring carboxyl group catalyzing the thermal reversible capability of another. This discovery is being explored further. Research on N-methylolacrylamide (NMA)-crosslinked cotton fabric, including hydrolysis studies with basic and acidic catalysts, has demonstrated that accessibility of catalyst is a critical limitation to crosslink reversibility. Creases durable to laundering have been ironed into NMA-treated fabric using sodium carbonate as catalyst, but similar treatment failed to remove the creases completely, apparently because the catalyst is inaccessible to some of the crosslinks. The significant contribution of basic catalysts to the thermal degradation of cellulose is being delineated by use of a differential scanning calorimeter, differential thermal analysis and thermal gravimetric analysis. From past and current results, it is becoming increasingly evident that the course of a crosslinking reaction and the reversal of this reaction are subject to limitations imposed by the cotton cellulose environment in which the reactions occur. (S2 1-261).

The grantee (Textile Research Institute) has conducted further exploratory work on the crosslinking of chemically modified cotton to obtain fabrics with an optimum combination of resiliency and thermplasticity. Because diester crosslinks provide unambiguous knowledge of the structure, nature, number, and length of crosslinks introduced, dibasic acid chlorides were investigated as crosslinking agents for cotton. Treatment conditions required, and the effect of variables in the preparation of crosslinked cottons with succinyl (C_4), adipyl (C_6), and sebacyl (C_{10}) crosslinks, were determined. Benzoylation of the diester-crosslinked cotton fabrics to levels

of substitution required for imparting thermoplasticity severely reduced the wrinkle resistance of the fabrics. Substantial degrees of thermoplasticity and wrinkle resistance were produced in fabric samples by resin treatment of partially saponified benzoylated cotton. The thermoplasticity of highly benzoylated cotton, degree of substitution (DS) 1.7, is not lost upon saponification, even to low DS values. Resin treatment imparts wet and dry wrinkle resistance with little diminution of thermoplastic crease retention. The resin treatment of partially saponified fabric, rather than fabric directly benzoylated to an equal DS, yields a better combination of wrinkle resistance and thermoplasticity. The fundamental chemistry of benzoylation and carbanilation is being elucidated to provide guidelines for further research. (S2 1-240(Gr.)).

Promising findings from completed exploratory research on selected cellulose-epoxide and cellulose-halohydrin reactions are being applied and extended in a study of reactions between cotton cellulose and various types of heterocyclic compounds to develop improved textile products. Various aminized cottons have satisfactorily catalyzed the opening of epoxide rings in a number of epoxides to bring about their reaction with the aminized cottons without use of external catalysts. Aminized cottons possessing tertiary amino groups have been quaternized with epichlorohydrin to produce cottons possessing high dry as well as wet crease resistance. Those possessing primary or secondary amino groups reacted with epichlorohydrin to produce cottons possessing high wet crease resistance only. Chemical modification of cotton with epichlorohydrin in the presence of various amines has resulted in both weak and strong anion exchangers possessing different fabric properties. Cotton has also been reacted with the heterocyclic compound 1-chloro-2,3-epithiopropane and its precursors. Introduction of both chlorine and sulfur into the modified cotton offers a tool for analysis and possibly for elucidation of reaction mechanisms. (S2 1-216; S2 1-282).

Cooperative research with the International Lead Zinc Research Organization involves treatment of cotton with finishes containing lead and other metal compounds to impart improved properties for specific end uses. In recent work, cotton printcloth was successfully impregnated with metallic lead on a semipilot-plant scale. Treatment with lead acetate followed by reduction with sodium borohydride was utilized. The metallized fabric possesses some flame and rot resistance, and also has potential for use in sound attenuation, radiation shielding and other applications. Thiomethyl-, thioethyl-, and thiopropyltriphenyllead all impart good rot resistance to cotton fabrics. Fabrics treated with 5% thioethyltriphenyllead have retained 100% of their strength after 23 weeks of soil burial. Since this finish does not impart color to fabric or reduce the strength of the fabric when applied, it offers some commercial promise. Duck and printcloth samples treated with this finish are currently under evaluation for weather-, mildew-, and rot resistance. Triphenyllead laurate, triphenyllead acetate and N-(tributylplumbyl)imidazole also show promise as finishes for imparting rot resistance to cotton. A more comprehensive study of the better finishing agents will be undertaken, and screening of new lead compounds will be

continued. (S2 1-232; S2 1-292).

Additional exploratory research on the chemical modification of cotton fabrics using reagents in the form of fogs or aerosols has been conducted. Ultrasonic fog generating equipment has been procured and test operated to fog dyes onto cotton fabrics. The degree of penetration of dyed water into scoured and bleached cotton twill fabrics when these fabrics have been frozen at varying moisture contents has been explored on a preliminary basis using air-type spray guns with high air dispersion rates. The rate of subsequent thawing as a controlling variable in degree of penetration of the treating media into the cotton has also been explored. Preliminary results are sufficiently promising that the techniques will be studied using thickening agents, anionic and cationic treating solutions, and thermoplastic emulsions as controlling variables for the penetration. (S2 1-247).

In contract research at Gagliardi Research Corporation, two promising procedures have been developed for improving wrinkle resistance and abrasion resistance of cotton fabrics crosslinked by reagents in the vapor phase. In one procedure, fabrics preimpregnated with nitrogenous compounds, such as urea, ethyleneurea, or hydroxyethyl carbamate, are crosslinked with vapors of methyl formcel-formic acid. Methyl formcel, a hemiformal solution of formaldehyde in methanol, has been found to have many advantages over paraformaldehyde and aqueous formaldehyde for vapor phase reactions on cotton. Among these are greater reactivity at milder process conditions and improved uniformity of reaction. In the second procedure, cotton preimpregnated with DMEU or other types of acid-curing cellulose crosslinking agents is treated with vapors of chlorosilane monomers. As the chlorosilane monomer polymerizes onto the cotton, evolved hydrochloric acid crosslinks the nitrogenous resin. These vapor phase grafting and crosslinking processes result in cotton fabrics and garments with high wrinkle resistance and significantly greater resistance to abrasion than similar products prepared by conventional pad-dry-cure processes. Durability of these properties to laundering is good. The potential of these vapor phase processes for preparation of durable press garments is such that many manufacturers are making detailed inquiries into the processes and a few manufacturers are now undertaking the design of appropriate commercial equipment for vapor phase processing. (S2 1-231(C)).

Continued good progress has been made in the research to improve resistance to edge abrasion damage in wash-wear cottons, particularly durably pressed garments, by application of selected polymers in conjunction with crosslinking agents. Two polymers show particular promise. A type of polyurethane capable of further polymerization has been found which, when used in conjunction with crosslinking agent, gives significant improvement in wrinkle recovery over that obtained with the crosslinking agent alone. The polymer also improves crease retention and permits use of about one-third less of the crosslinking agent. Abrasion resistance as measured by laundering is improved at least twofold. Another promising polymer is an imine terminated polyether. This material in conjunction with crosslinking agent gives substantial improvement in conditioned and wet wrinkle recovery,

tearing strength, and moderate improvement in breaking strength. A variety of polymer additive treatments are currently being wear tested on all-cotton shirts and trousers. A second approach investigated in the research has been the combination of polymer additive treatments and differential crosslinking. In general, use of polymer additives has given significant improvement in wrinkle recovery, crease sharpness and fuzz resistance over that obtained by differential crosslinking alone. The abrasion resistance of cotton goods finished with these combination treatments is several times that of conventionally crosslinked fabric. (S2 1-260).

The contractor (Southern Research Institute) has conducted further exploratory work on the use of reactive swelling agents to improve moisture absorptivity of wash-wear cotton fabrics. Several promising approaches for achieving high moisture absorptivity and wrinkle recovery have been discovered but additional research, both in application and evaluation, will be required. Cotton treated with dimethylolethyleneurea in the presence of benzyltrimethylammonium chloride had excellent crease recovery properties and moisture absorptivity equal to that of the untreated cotton. Cotton treated with methylolated urea-N,N'-bis(N-methyleno-N-methylmorpholonium iodide) had a higher moisture regain than the untreated cotton. Cotton fabric treated with mixtures of dimethylolethyleneurea and bis(N-methylol-2-carbamoylethyl)dimethylammonium iodide or ethylene bis(oxymethylpyridinium chloride) had high crease recovery and moisture regain values. Coapplications of trimethylolmelamine with polyoxyethylene glycols at a level of 200% of the weight of the trimethylolmelamine gave treated fabrics with moisture regains similar to that of untreated fabric. (S2 1-239(C)).

Contract research recently initiated at Harris Research Laboratories, Inc., has as its objective the development of finishes for cotton fabrics to render them more rapid drying. In basic studies, the water content of wet cotton fabrics before and after centrifugation was found to vary greatly with fabric construction. The loftier fabrics retained the most water. The time required for drying, however, was found to depend not only on water content but also on fabric construction as an independent variable. Drying time appears to be a function of the water bound by capillary action within the fabric structure. Application of 2% of a commercial fluorochemical water repellent in combination with standard commercial wash-wear agents decreased the water content of 8-oz. duck after centrifugation by as much as 65% and decreased by more than 50% the time required for tumble drying. Wash-wear agents by themselves caused only a minor decrease in the water content. A search will be made for ways of decreasing drying time of cotton fabrics without rendering them water repellent. (S2 1-243(C)).

A fundamental study of the preparation and properties of phosphonitrilic and phosphoryl chloride derivatives having potential for reaction with cotton--conducted under a P. L. 480 grant at Birkbeck College of University of London--is now nearing completion. The research stemmed from work completed under an earlier P. L. 480 project UR-E29-(20)-35 in which the chemistry of these interesting inorganic compounds was placed on a sound, systematic basis. A large number of compounds of this type have been prepared and

characterized. Details of the synthesis, separation and purification of these compounds have been the subject of some 20 publications. Mechanisms of the reactions and rearrangements of many of these have been elucidated. Some of the compounds have configurations that suggest ability to react with cotton cellulose or for attachment of side chains that will afford reactivity. Fundamental information of this type will provide useful leads for the development of practical treatments for cotton to afford improved properties, such as flame resistance, ablative and wash-wear properties. (UR-E29-(20)-55).

2. Chemical Reactions Initiated in Cotton Cellulose and Chemically Modified Cotton by High-Energy Radiation, Light, and Heat. Research on gamma radiation-induced reactions of cotton is leading to the production of cotton textiles with new or improved properties. Radiation grafted vinyl-cotton copolymers are currently being studied. Polyacrylonitrile-cotton copolymers have shown the most interesting changes in textile properties and are thus being most extensively investigated. It has been shown that changes in flat and flex abrasion resistance and some other textile properties are dependent upon the experimental conditions of the grafting reaction. In fabrics grafted with polystyrene, marked changes in properties were observed at a graft polymer content of 42%. Of particular interest was a large increase in flex abrasion resistance. At even higher polystyrene contents (67%), extremely high flex abrasion resistance was obtained (approx. 10,000 cycles). Yarns and fabrics grafted with polymethyl methacrylate have been prepared by a simultaneous radiation technique and the textile properties of these copolymers will be evaluated. Satisfactory conditions for grafting of polyvinyl acetate to cotton by the simultaneous radiation technique have not yet been found. Further work will be carried out because of the interesting results obtained with this polymer using the post-irradiation grafting technique. (S2 1-195(Rev.)).

The localization of energy that is deposited within the cotton cellulose molecule from incident energy, such as heat, light, weathering, mechanical action, chemical action, or ionizing radiation, generally leads to changes in the properties of cotton which are not always desirable. Basic processes for directing energy transfer within the cellulose molecule to minimize these undesirable changes are being investigated. Research has shown that substitution of benzoyl groups on the cellulose molecule protects fibrous cotton cellulose from degradation by weathering or exposure to high-energy radiation. It has been experimentally demonstrated that free radicals are formed in fibrous cotton cellulose on absorption of energy from photochemical, radiochemical, and thermal sources. By means of electron spin resonance (ESR) spectra, the role and kinetics of radical ions in reactions initiated by light in fibrous cotton cellulose and related model compounds have been determined. Semiquinone radical ions in the photosensitized oxidation of alcohols have been related to the photodegradation of cotton cellulose sensitized by anthraquinonoid vat dyes. The role of free radicals in ceric ion initiated graft copolymerization reactions of cotton cellulose with acrylonitrile has also been demonstrated. Interpretation of the effect of temperature on the formation of these free radicals gave apparent

activation energies for radical formation on cotton cellulose as 34 kilocalories per mole. The ESR spectra of gamma-irradiated cotton cellulose I and II showed that the change in lattice type involves a rearrangement of the hydrogen bonds within the unit cell. (S2 1-270).

3. Mechanisms, Rates and Catalysis of Reactions of Cotton Cellulose and of Chemically Modified Cotton. In recent studies of mechanisms of cellulose etherifications, the rates of reaction between cotton and the following compounds in the presence of four inorganic salt catalysts have been determined: (1) dihydroxyethyleneurea, (2) dimethyloldihydroxyethyleneurea, (3) dimethylolpropyleneurea, and (4) 1,3-dimethyl-4,5-dihydroxy-2-imidazolidinone. Several metal salt complexes of propyleneurea have also been isolated and characterized. Currently used delayed cure processes for cotton are based on differences in rates of condensation and differences in rates of etherification of cotton with the various N-methylol compounds under investigation, and the research is providing knowledge of the importance of the catalyst on these rates and basic information as to types of bonds the metal ion makes with the urea portion of the finishing agents. As an aid to elucidation of mechanisms of crease resistance in cotton, the effects of post-mercerization on the fine structures of cottons etherified with d,l-isomers of butadienediepoxide under conditions of aqueous basic catalysis have been determined, and several quaternary cellulose anion exchangers in fabric form have been prepared and characterized. (S2 1-277).

An investigation of mechanisms involved in producing dry and wet crease resistant cottons by esterification with derivatives of monobasic acids is in progress. Earlier work established that cotton can be esterified with selected monobasic acid chlorides in nonaqueous media, such as dimethylformamide, to produce partial esters of low degree of substitution which have high dry and wet crease resistance. Some preliminary work on methods of esterification other than the dimethylformamide-acid chloride method has now been completed. Partial esters prepared by various methods will be evaluated to determine effect of solvent, mechanism of esterification, and other experimental parameters on the fabric properties. (S2 1-233; S2 1-294).

A study of the oxidation of cotton and crosslinked cotton by hypochlorite, hypobromite and other agents commonly used in bleaching of cotton products is being conducted at the Institute for Fibres and Forest Products of the Ministry of Commerce and Industry of the State of Israel. The P. L. 480 project under which the work is being done is an outgrowth of an earlier project, UR-A10-(20)-4, now completed. Basic information is being developed concerning the conditions governing the oxidation, degradation and yellowing effects that occur on mild oxidation of cottons treated with crosslinking agents commonly used in the easy-care finishing of cotton fabrics. Oxidation studies have shown that crosslinked cottons have higher chemical stability than noncrosslinked cottons. Functional groups formed during oxidation of crosslinked cottons give some indication of the sites of attachment of the crosslinking agent on the cellulose molecule and may be useful

in determining the average distance between crosslinks. The information obtained in this research is expected to be useful in improving the characteristics of cottons, especially fabrics treated for easy-care properties for various end uses. (UR-A10-(20)-50).

4. Investigations of Soiling and Soil Removal from Cotton Textiles. Additional fundamental information on soiling and soil removal characteristics of cotton finishes has been obtained by the contractor (Harris Research Laboratories, Inc.). Experiments with fabrics selected to represent the major classes of surfaces indicated that, in drycleaning solvent, the factor having the greatest effect on soiling and soil removal was the nature of the coating on the fabric. Fabrics with applied coatings soiled much more than uncoated or unmodified fabrics. Fabrics having acrylic or silicone coatings soiled less than those coated with fluorocarbon. Fabrics crosslinked with formaldehyde soiled more than noncrosslinked fabrics. Form D fabrics (crosslinked in the nonswollen state) soiled more and were more difficult to clean than Form W fabrics (crosslinked in the swollen state). Attachment of charged groups to the finish or to cotton by chemical modification increased the degree of soiling. Generally, less soil was removed by the drycleaning process than by laundering. In the case of soiling in air, there is very little difference between practical vacuum cleaner dirt soil and iron oxide model soils in soiling and soil removal properties. In drycleaning medium, however, fabrics soil much less with the vacuum cleaner dirt than with either dry or oily iron oxide soils and the dirt is easier to remove by drycleaning. This indicates that there may be a selective interaction between the soil components and the fabric surfaces, and that the properties of the medium affect the fabric-soil adhesion. The hardness of the applied finish appears to have a definite but minor effect on soiling in air or in aqueous media, and little or no effect in drycleaning medium. Apparently the very low force of impact associated with soiling in drycleaning solution as compared with that for the other media produces a different degree of soiling, explaining this behavior. Knowledge from this research can be useful in the development of better soil resistant finishes for cotton and possibly in techniques employed for cleaning fabrics. (S2 1-223(C)).

In grant research recently initiated at the University of Arizona to determine the surface microstructure of untreated and chemically finished cottons with relation to soil attraction and soil retention, methods have been developed to permit preparation of replicas of soiled cotton samples for electron microscopical studies at magnifications greater than those heretofore employed. Equipment is being constructed to provide cold incineration (oxidation by activated oxygen) of specimens of soiled cotton for studies with the electron probe. The probe technique should provide a unique tool for analyzing the elements in the soil particles. Basic information on the nature of contaminating particles, of the fiber surface, and on the mechanism of soil adhesion should point the way to improvements in soil resistance of cotton textiles. (S2 1-238(Gr.)).

5. Exploratory Physical Investigations of Native and Modified Cotton. Basic investigations of tensile and torsional properties of crosslinked and other types of modified cottons have continued. Measurements of tensile strain recoveries of single cotton fibers after three minutes' relaxation under standard atmospheric conditions showed that fibers crosslinked by various formaldehyde and DMEU treatments display recoveries of the same ranking as the corresponding treated yarns and fabrics. Crosslinked fibers also display wet recoveries (from strain corresponding to a 1-gram load) with the same ranking as the corresponding yarns and fabrics. Tensile strain recoveries of both the standard conditioned and wet fibers increase as the number of crosslinks increases. The tensile recovery of single fibers may also differentiate the type of formaldehyde treatment, even with the same amount of crosslinks present. Single cotton fibers specially crosslinked during sulfuric acid swelling were found to have large increases in wet recovery. It was found that breaking strengths for wet single fibers are reduced by chemical modifications, as is the case with yarns and fabrics. However, the decrease in modulus due to water and the increase in modulus due to crosslinks are both much less for the fibers than for corresponding yarns and fabrics.

In further studies of torsional properties, the frequency of recovery from an induced torsional deformation has been determined for single fibers under dry, standard, and wet conditions. Even with the high variances found, the increase in recovery for pad-dry-cure cottons is better than onefold greater than that observed for untreated samples. Torsional deformations due to wetting are completely reversible for mercerized and mercerized-crosslinked cottons. The resultant inverse relation between rotational amplitude and rotational frequency for single fibers agrees with the relation between stiffness and recovery for yarns and fabrics. Rotational deformation due to sodium hydroxide swelling of single fibers is not completely recoverable by water washing and drying. The recovery of untreated or resin treated fibers from bending has been found to decrease as the angle of bend is increased, and to increase as the resin add-on is increased. The basic information being developed will aid in devising means for improving properties and performance of the modified cottons. (S2 1-262).

To better characterize and compare chemically modified cotton yarns and fabrics, and thereby facilitate the development of improved products, an investigation of the tensile recovery behavior of cotton yarns and fabrics under different conditions and methods of testing has been initiated. Yarns were found to have better recovery at low extensions than the fabrics from which they were removed, but the yarns' recovery decreased more rapidly with increases in extension. Yarns treated with urethane had better recovery than the untreated yarns but considerably poorer recovery than resin treated yarns. When ply yarns of different sizes were compared, the finer yarns showed better recovery. Mercerized fabric exhibited better recovery than unmercerized. With resin treated fabrics, a series of recovery measurements made on the same specimen did not give the same recovery values as obtained by the conventional procedure where separate specimens are tested. Energy

measurements and their relation to tensile recovery determinations will be investigated. (S2 1-285).

For research studies of the relation of fiber properties to physical behaviors of mechanically and chemically treated cotton fabrics, printcloth fabrics of comparable structures made from Deltapine, Hopi Acala, and Pima cottons have been treated in scoured and in slack mercerized form to resin add-ons of 2.5 and 6% dimethylolethyleneurea (DMEU). Significant increases in abrasion resistance, as determined by both the Stoll Flex Abrader and the Accelerotor, resulted from the slack mercerization treatment. The abrasion resistance was decreased by the resin treatment, the decreases becoming larger as the add-on was increased. Significant differences were observed among the Deltapine, Hopi Acala, and Pima fabrics--the abrasion resistance of the Pima cottons was greater until the add-on reached 6%. At this add-on no differences among the cottons were obvious. Additional analyses are being made to establish the reliability of this research finding that the cottons which differed as untreated, scoured, and mercerized fabrics reach a comparable abrasion resistance level when the DMEU add-on is high. Tension during the resin treatment as well as tension on the scoured and slack mercerized controls decreased the abrasion resistance. The increase in swelling of cotton in 6 to 8% sodium hydroxide by dilution after treatment with the normal 25% sodium hydroxide has been confirmed by the ACV test. A contraction in yarn length, an increase in yarn strength, and an increase in moisture regain of the cotton also result from this process. Cottons with different fiber properties differed in their swelling behaviors. Other work has shown that the strength, elongation, modulus, and toughness of the individual fibers correlate highly with these respective properties measured on fabrics of widely varying treatments. This indicates that success in development of fabrics of greater serviceability will probably be achieved principally by processes that improve the fibers. The production of a satisfactory textile product from fibers of low performance characteristics will be very difficult. (S2 1-272).

In the research to determine the simultaneous effect of pertinent fiber properties and combinations of fiber properties on yarn properties and spinning performance to provide guides for obtaining maximum utilization of cottons of varying fiber properties, fifty-one medium staple cottons have been processed and spun into yarns. Several preliminary findings have been obtained by evaluation of fiber, yarn and end breakage data from twenty-eight of these cottons. It has been found that fiber strength contributes most toward yarn strength, and fiber length contributes most toward end breakage. Fifty percent span length is a better basis to use than the customary 2.5% span length in explaining the effects of fiber length on yarn properties and end breakage. Single strand strength variability is the most important yarn property affecting end breakage. On an average, about 88% of the factors which contribute to yarn strength and 56% of the factors which contribute to end breakage may be attributed to fiber properties. Yarn elongation is linearly related to yarn size expressed in yarn number. It was found that the roving twists required

for maximum spinning performance differed widely for the various medium staple cottons, and these roving twists were directly proportional to the yarn twists required for maximum strength. A technique was developed for predicting yarn strength on an absolute basis from fiber strength, fineness and length distribution of a cotton. Also, means appear to have been found to enable the theoretical substitutions of one fiber property for another while maintaining yarn skein strength constant. (S2 1-207).

Further experiments by the contractor (Macrosonics Corporation) have clearly established that treatment of cotton samples in a "cavitating" field in distilled water by irradiation with acoustic energy causes major fiber modification. Treatment of both Acala 4-42 irrigated and Deltapine raingrown cottons at energy levels of 20 watts/liter in the 20-40 KCS frequency range produced intense fibrillation, fiber matting, and a peculiar crossmarking phenomena observable under polarized light. Apparently, cotton is sensitive to specific sonic frequencies, since no physical changes were observed at other frequencies and intensities. Fibers cut to 1/8-inch-1/4-inch lengths and treated at 20 watts/liter and 30 KCS produced hand sheets of paper of acceptable uniformity and with up to 56% of the strength of standard high-strength papers. The application of acoustic energy thus may have value for the utilization of short cotton fibers and linters in producing high grade and specialty papers. Airborne irradiation of Deltapine and Acala 4-42 cottons at high intensities (160 db) and low frequencies (30 KCS) showed that no significant physical or chemical modification of the fibers occurs before reaching the decomposition point. These tests indicated the importance of the apparent density of the cotton in sound-degradation phenomena. The importance of frequency with relation to irradiation in air has not been entirely clarified. (S2 1-222(C)).

Under a P. L. 480 project at the Chalmers University of Technology in Gothenburg, Sweden, a basic investigation of the behavior of cotton fibers when subjected to aerodynamic forces is being carried out. Apparatus has been designed which permits the taking of high-speed motion pictures of separated cotton fibers traveling in air streams of different velocities. Conditions and designs are being studied which will cause the fibers to become parallelized through aerodynamic forces exerted by the flowing air stream. Studies conducted in air streams of relatively low velocity have provided much useful information concerning the behavior of cotton fibers under these conditions. In its later stages the research is moving into studies in the area of higher velocity air streams. Fundamental information of the type being developed in this research is prerequisite to the design considerations in devising totally new and unorthodox methods for the processing of cotton into useful textile products. (UR-E26-(20)-6).

An investigation of means to minimize fiber hooked ends in cotton card and drawing slivers is in progress under a P. L. 480 project at the Ahmedabad Textile Industry's Research Association in India. End breakage in the

processing of cotton is related to the presence of hooked ends in the fibers making up sliver. Conventional processing organizations tend to remove hooked ends, but in abridged processes such as direct spinning, the fewer drafting processes between carding and spinning allow more fiber hooks to remain and hence adversely affect spinning efficiency, since fibers with ends hooked do not make their full potential contribution to strength and other characteristics of the yarn assembly. Observations made concerning the effect of relative speeds of machine elements in the card and sliver size may offer clues to the physical mechanism by which fiber hooks are formed. Basic information obtained in the work under this project is expected to be of use in facilitating direct spinning and improved cotton processing through means to minimize the amount of fiber hooks in card or first drawing sliver. (UR-A7-(20)-51).

Research is in the early stages under a P. L. 480 project at the Shri Ram Institute for Industrial Research, Delhi, India, in which an investigation is being conducted of heat and mass transfer rates and other engineering concepts as related to the drying and curing of resin-treated cotton textiles by countercurrent solid-gas contact systems. Fluidized bed techniques provide highly efficient means for effecting heat transfer in many engineering applications. The basic engineering data being obtained in this research is prerequisite to adapting this system to the drying and curing operations in textile processing. If successful, the adaptation of the fluidized bed technique will be of great practical value in increasing efficiency and lowering costs in cotton textile finishing. (UR-A7-(20)-84).

C. Technology--Process and Product Development

1. Improved Procedures for Mechanical Processing of Cotton. The research investigation of blending methodology to establish optimum blending procedures for maximum utilization of cottons differing widely in fiber properties has been completed. Studies of the relation of spinning efficiency to prespinning processes and to fiber properties indicated that the need for good blending prior to picking is most critical for cottons differing in bundle tenacity; less critical for cottons differing in fineness; and least critical for cottons differing in short fiber (3/8" and shorter) content. Mathematical techniques showed that for two stages of drawing there was no significant fiber breakage for length groups shorter than the roving roll setting of 20/16", but significant breakage (40-50 percent) of the longer length groups (21/16" and 23/16"). For no drawing stages, fiber breakage of the longer fibers was much less, indicating that when fibers are straightened as a result of drawing, breakage at the roving frame is more severe. Progress was also made in the development of a theoretical approach for interrelating length distribution, yarn count, and fiber fineness with spinning performance. Information from this research will enable selection of the correct blending procedures in relation to fiber properties, thus facilitating the production of products of improved quality from cottons differing widely in fiber properties. (S2 1-234).

Research was recently initiated to determine the interaction of processing variables with yarn properties and end breakage in spinning at high processing speeds, to produce cotton products of required properties at lowest cost. Guides will be developed for selecting the combination of spinning variables needed to most economically produce yarns having the properties required for specific end uses. Selected cottons suitable for use in the research are being obtained. Meanwhile, preliminary experiments at selected stages of processing are being conducted to ascertain which processing variables are the most important for initial study. The Uster Spectrograph, an instrument for characterizing defects in cotton products at different stages of processing, is being evaluated to determine its potential usefulness in the research. (S2 1-295).

The contractor (Auburn Research Foundation) has conducted further work to determine optimum processing procedures for cottons differing in tensile and elastic properties, and relate these properties to mechanical processing performance and yarn and fabric properties. The processing performance evaluations of the 1-1/16" cottons differing in fiber bundle elongation have been completed. Generally, the higher elongation cotton spun with fewer ends down than the lower elongation cotton, this effect being more pronounced at higher drafts, lower twists, higher spindle speeds, and as the yarn number became finer. An increase in fiber elongation from about 6 to 10% made possible an increase in spindle speed of about 1000 for the high twist 30/1 and 40/1 yarns at constant end breakage rate. No relation between fiber elongation and processing performance prior to spinning was found. Single strand yarn strength and yarn elongation were directly and linearly related to fiber elongation. Replicate number 1 of processing performance evaluations for 31/32" cottons having high and medium elongation has been completed. In addition to further evaluations of 31/32" and 15/16" cottons, work will be initiated on a 25,000 spindle hour spinning test conducted under commercial conditions. (S2 1-242(C)).

Good progress has been made in the research to determine the effect of high production carding on fiber length distribution and fiber hook formation in card sliver, and to establish improved drafting procedures required for maximum removal of fiber hooks for carded and combed yarns. Results of tests with a 1-1/16" irrigated Acala cotton showed that increased carding rate caused the majority hooks to decrease and the minority hooks to increase, as previously found for a 1-1/16" Deltapine cotton. The rate of increase for the minority hooks was greater and the rate of decrease for the majority hooks was less than found for the raingrown Deltapine cotton. End breakage for the lots of cotton processed using the SRRL recommended drafting directions (i.e., majority hooks trailing at first and second drawing, leading at roving, and trailing into spinning) was less than for those processed using conventional drafting directions. High carding rate resulted in fewer noils being removed at the comber, probably as a result of the decrease in the majority hooks with high carding rates. The rate of increase in yarn imperfection count with increased carding rate was greater for the irrigated Acala than the raingrown Deltapine; however, for the

combed yarns there was only a slight increase with increased carding rate. The research findings from current and earlier investigations on fiber hooks and drafting directions required for maximum hook removal and processing efficiency have been evaluated commercially with promising results. (S2 1-274).

2. New and Improved Mechanical Processing Machinery--Opening Through Carding. Construction of the full-size, mill-type unit of the SRRL Bale-Opener-Blender has been completed. Based on results of laboratory tests of the unit, certain modifications were made to achieve the desired performance. The machine will now be evaluated extensively under commercial mill conditions. The Blender makes possible for the first time the opening and homogeneous blending of cotton from as many as 20 bales within a single machine. It processes a continuous sandwich bale at controlled production rates up to 1,000 pounds per hour--about three times the production of conventional "blenders." In contrast to conventional blenders, where reliance for blending is placed on subsequent processes, the new machine delivers thoroughly blended cotton in any desired proportion. Another advantage of the Blender is that it will require only half the floor space of equipment now used to open, or fluff, and blend the tightly packed cotton as received at the mill. The Blender should fill the urgent need in the textile industry for improved blending of cotton. By efficiently blending many bales in the initial opening process, it will be possible to average out fiber differences within and between bales and obtain the stock uniformity necessary for high processing efficiency in today's modern mills. (S2 1-252).

Research to develop and evaluate prototype equipment for feeding cotton to textile cards to produce cotton products with improved physical properties is continuing. Further work was conducted to devise a suitable method for feeding to textile cards the drafted lap produced by the recently developed Lap Drafter. Research with a bench model machine has shown that a drafted lap can be processed satisfactorily by using a small diameter, high-speed precision cylinder with a unique tooth design and an enclosure with specially designed openings for introducing the drafted lap, extracting trash, and removing the fiber. Cotton lap processed through this apparatus has the openness of a card web but a higher nep count. Tests indicate exceptionally good trash extraction. Construction and installation of a full-size apparatus on a textile card is almost complete. Its performance, independently and in combination with the Lap Drafter, will be evaluated. Preliminary research is also being done to establish a basis for developing a mechanism to feed multiple laps of loose cotton to the lickerin of the previously developed Precarding Apparatus. Four thicknesses of lap cannot be fed satisfactorily with a conventional mechanism; however, encouraging results are being obtained with an experimental mechanism. The development of improved systems for feeding cotton lap and loose cotton to textile cards will aid in automating this important stage in textile processing and in improving the quality of cotton products. (S2 1-279).

In further research on short fiber removal at the card, evaluation of the

experimental mechanical unit for combing the output of the card to improve fiber parallelization and to remove short fibers has revealed no significant improvements in the card sliver. Extensive modifications of this unit are being made and it is expected that testing will be resumed soon. For efficiently evaluating the unit's ability to improve fiber alignment in the sliver, a special clamping device based on the Lindsley technique has been constructed. In another approach to the objective of this research, a basic investigation is underway to develop a means for removing the aligned fibers from an electrostatic field in the form of an aligned strand. Such a device could be combined with the card to remove the short fibers, parallelize fibers, and form a strand. Initial research is being conducted with the strand forming device in combination with a special devised feed system and the electrostatic fiber fractionator developed under an earlier project. Preliminary testing has produced short lengths of highly fiber-oriented strands from raw cotton. However, the strands are nonuniform in appearance, and production rate is low. (S2 1-273).

The contract research at General Applied Science Laboratories, Inc., on aerodynamic separation of lint cotton into individualized fibers has been successfully completed. Recent work has involved a comprehensive analytical investigation of the dynamics of cotton fibers and clumps in corner flow, and a theoretical treatment of the effects of a dilation flow on an idealized fiber tangle. The valuable fundamental information developed under this project has suggested several areas of promising research which are more directly pointed to specific applied problems on developing new and improved textile processing machinery. A more comprehensive study of the application of dilation force fields to open lint cotton into individualized fibers is considered particularly desirable. (S2 1-204(C)).

Limited progress has been made in a recently initiated investigation of fundamental actions of cotton textile processing equipment by means of high-speed photography to obtain information needed to develop radically new processing machines and to improve conventional machines. Analysis of high-speed motion pictures made in an investigation of the action of the card doffer comb did not give any significant results. In order to more efficiently conduct this highly specialized line of research, professional personnel will be trained in special techniques required to make time-space analyses by high-speed photography and other means. The research will be held in abeyance until that time. (S2 1-278).

3. Wash-Wear Cotton Textiles and Garments. In research to produce improved wash-wear cotton through swelling treatments, it has been found that swelling of cotton with phosphoric acid or sulfuric acid before dry-cure crosslinking improves wash-wear performance mainly on light fabric that otherwise performs poorly. Wash-wear ratings, particularly of line-dried fabrics, are increased as a result of the swelling treatments. To be of any benefit the swelling must not cause decrystallization of the cotton. Swelling after crosslinking, by itself, does not improve wash-wear performance. Modification of the cotton cellulose by pretreatment with an agent containing

noncrosslinking substituents, especially those that contain a hydrophilic group such as the hydroxyethyl, can be as effective as a crosslinking modification as an adjunct to dry-cure crosslinking. The noncrosslinking agents improve the wash-wear performance of the cotton fabrics, and generally cause less reduction in strength and abrasion resistance of the fabrics. The effectiveness of noncrosslinking substituents in improving wash-wear performance with dry-cure crosslinking supports the proposal that the primary effect of crosslinking substituents introduced with swelling is to preserve swelling. Emphasis in new work will be on the improvement of abrasion resistance by crosslinking of cotton in the partially swollen state. (S2 1-235).

Investigation of the crosslinking of cotton with new types of N-methylol and related derivatives to produce smooth-drying, wrinkle-resistant fabrics has continued. Current emphasis is on stable crosslinking agents suitable for use in delayed-cure processes. By insertion of carboxylic acid groups and phosphonic acid groups into N-methylolamide crosslinkers also containing tertiary amino groups, agents with improved reactivity to cotton have been produced. Insertion of an ester group into an N-methylolamide crosslinking agent has been found to improve the resistance of the finished fabric to chlorine damage. Presumably, this desired effect is obtained after partial or total hydrolysis of the ester group. The synthesis of β,β'-iminobispropionamide--a useful intermediate for preparing new crosslinking agents--has been improved. The N,N-bis(methoxymethyl) derivatives of acetamide and ethyl carbamate appear to have potential for use in deferred curing. Satisfactory procedures have been developed for synthesizing 1-hydroxymethyl-3-methyl-4,5-dihydroxy imidazolidinone, a potentially valuable delayed-cure crosslinking agent. Preliminary experiments on delayed-cure processing of cotton fabric with this agent showed that after 21 days of storage of the treated, uncured fabric it could be cured to give a wash-wear fabric with high dry wrinkle recovery and moderate wet wrinkle recovery. Little if any formaldehyde odor was given off from the treated fabric. Evaluation of a commercially available diisocyanate, methylenebis(4,4'-cyclohexylisocyanate), as a wash-wear agent showed that treatment of cotton fabric with a dimethyl formamide solution of this chemical imparted many desirable properties. The treated fabrics had good wrinkle recovery, were resistant to acidic and basic hydrolysis, exhibited no strength loss due to chlorine damage, were stable to the wash test and were not discolored by bleaching, and showed resistance to rot. When octadecylisocyanate was incorporated into the pad bath, fabric tearing strength and abrasion resistance were increased. A more practical method for the application of diisocyanates to cotton fabrics will be sought. (S2 1-227; S2 1-289).

The research on carbamate finishing of cotton is currently directed toward developing new and modified finishes for deferred-cure processing to yield chlorine-resistant, lightfast fabrics. Further study of dimethylol ethyl carbamate has shown that with this agent very short curing times (as little as 20 seconds) at 200°C can produce good finishes if magnesium chloride is used as catalyst. Treatment of cotton with dimethylol allyl carbamate

gives wrinkle-resistant, chlorine-resistant fabrics that contain olefinic linkages capable of further chemical reaction. Dimethylol carbamoyloxyethyl phthalate and dimethylol benzoxyethyl carbamate have been used as finishing agents to produce wrinkle- and chlorine-resistant cotton fabric. Methyl carbamate-finished cottons which also are wrinkle- and chlorine-resistant were produced effectively by curing the fabrics between metal plates for 2.5-15 seconds at 200-220°C. Dimethylol hydroxyethyl carbamate, a new agent developed in this research, is now being used commercially for the production of wash-wear, durably pressed cotton goods. It is a better deferred-cure agent than the dimethylol monoalkyl carbamates that have been employed in commercial wash-wear finishing. A new group of acid catalyzed, reactive dyes for cotton have been prepared by reacting hydroxyethyl carbamate with certain vinyl sulfone or chlorotriazine dyes and methylolating the products. The dyes may be reacted simultaneously with crosslinking agents to impart wash-wear or durable press properties and color.

Investigations of the chloramide stabilities of carbamate-finished cottons are providing valuable new information on the chemistry of chlorine retention and scorch damage. The chloramide decomposition temperature of cotton finished with methylol isopropyl carbamate has been found to be about 30° higher than that of methylol methyl carbamate-finished cotton. Strength loss in the scorch test appears to be a function of the amount of chlorine released upon scorching. For cotton finished with dimethylol methyl carbamate, release during scorching of less than about 0.08% chlorine causes little damage, whereas above about 0.08% chlorine release, strength loss increases rapidly. The presence of moisture at the time of cure may affect unfavorably the chlorine resistance of cotton finished with carbamates by high-speed curing techniques. (S2 1-281).

Contract research at North Carolina State University, Raleigh, on stretching and compressive shrinkage effects in ease-of-care fabric treatments has been completed. Ethyl carbamate was found to be a good permanent resin for ease-of-care treatments, being superior to DMEU which had reduced effectiveness after commercial launderings and to APO which was considered unsatisfactory because of color problems. Mercerization, even though not reaching the level in elongation caused by slack mercerization in the laboratory, increased both tensile and tearing strength as did tension prior to curing. The semicommercial mercerization treatments employed in the project caused much smaller changes in fabric properties than had been found in laboratory treatments. Compressive shrinkage prior to curing caused marginal improvements in seam pucker, smooth drying properties, and some of the physical properties, whereas compressive shrinkage after curing had no effects on fabric properties. While the improvements found were considered marginal, the contribution of marginal effects could be sufficient to make previously unsatisfactory commercial products generally acceptable to the consumer. (S2 1-183(C)).

Research to develop wash-wear cottons of high abrasion resistance by the application of durable polymeric coatings has continued. Dimethyl silicone

of molecular weight 165,000 has been crosslinked as a film on cotton twill and sateen by benzoyl peroxide catalysis. The method has been adapted to permanent press techniques by using a hot-head press to simultaneously crease and cure the impregnated fabric. Efficiencies of 50-75% in insolubilizing the silicone were obtained by using pressing times of 1-2 minutes and a steaming time of 30 seconds. On sateen, crease recoveries of 265-275° dry and 255-269° wet were readily obtained by add-ons of 3-5%. Polybutadiene, polyethylene oxide (molecular weight, 600,000), poly(vinyl methyl ether), and copolymers of vinyl chloride with vinyl ethyl pinate and vinyl tetrahydroabietate have also been crosslinked as films on cotton fabric by curing with benzoyl peroxide. Crosslinked polymer films can also be used in conjunction with cellulose grafting or crosslinking processes to give wash-wear finishes. Application of a peroxide-crosslinked dimethyl silicone film on top of a grafted layer of a self-crosslinking polyacrylate resulted in higher wash-wear ratings than obtained by either treatment alone. Fabric to which a crosslinked film of dimethyl silicone had been applied was subsequently treated with APO in the absence of cellulose crosslinking catalysts. With 8% silicone and 3% APO applied, a wash-wear rating of 4.7 was obtained; this was higher than for either treatment alone. In another phase of the work, it has been found that cotton yarns with durable stretch properties can be readily prepared by applying certain crosslinked polymer films to false twisted yarns. A commercial self-crosslinking polyacrylic latex applied to coiled cotton yarn and heat-cured imparted a false twist durable to repeated flexing in both the wet and dry states; recoverable stretch was 105-125%. On mercerized coiled yarns, 165% stretch was noted. Crosslinked dimethyl silicone film applied to coiled yarn imparted 55% recoverable stretch. Grafting of the polyacrylate to cotton may account for the greater effectiveness of that polymer. Further research on development of polymeric finishes for cotton is planned. (S2 1-253).

Research was recently initiated to study the blending of treated and untreated cotton fibers as a means of improving abrasion resistance of wash-wear, durably pressed garments. In initial experiments, picker lap prepared from scoured and bleached rawstock was impregnated with Permafresh 183, a commercial delayed cure resin, to 18.1%, 12.1%, 8.8%, and 7.0% add-ons, dry basis, using immersion techniques. The dried but uncured fibers were then blended with scoured and bleached rawstock in the ratios of 75% to 25%, 50% to 50% and 25% to 75%, either at the picker or the drawing frame. No particular difficulties were encountered in carding, drawing, or roving. Sateen and twill fabrics were produced with a 50/50 blend in the warp and the above blends in the filling. Pressed, creased and cured sample cuffs made from these fabrics showed significantly better resistance to abrasion than cuffs made from control fabrics treated to corresponding add-ons in the piece. They averaged 22 launderings before any abrasive damage was noted, compared to 6 to 7 launderings for the control cuffs. Similar results were obtained in other experiments where blends containing one-third each of thermoplastic treated fibers (1-1/2 and 3% add-on of vinyl acrylate), thermoset treated fibers (7 and 18.1% add-on levels), and scored and bleached rawstock were evaluated in the filling.

The possibility of impregnating sliver with permanent press type resins followed by drying but not curing was also investigated. Blending of these slivers with untreated slivers from scoured and bleached cotton rawstock at the drawing frame resulted in less uniform rovings, insofar as distribution of treated and untreated fibers was concerned, than was achieved when blending was done at the picker. Fabrics have been produced for wash-tumble dry tests. The effects of thermoplastic-impregnated fibers, with and without catalyst, in three-way blends achieved partially at the picker and partially during drawing are being evaluated in wash-tumble dry tests. Extending the service life of permanent press garments by washing and tumble drying them inside-out is also being evaluated. Approximately 50 yards of 3/2 twill with 50% treated and 50% untreated fibers in warp and filling have been produced for a comprehensive study of variability of washing and tumble drying equipment available at the Southern Division and elsewhere. There is some indication that gas drying may be more damaging to durably pressed garments than electric drying. Sixteen pairs of work pants made from 50% blends (10% chemical add-on in the treated portion) are being in-service evaluated at the Southern Division. Preliminary results are encouraging. Simulated (approximately knee length) trouser legs with cuffs are also undergoing evaluation in wash-tumble dry tests. (S2 1-286).

Cooperative research with the Cotton Producers Institute to develop optimal cotton fabric structures for men's trousers and dress suits has continued. The study of the effect of yarn ply twist on wearability of seersucker fabrics woven from commercial tension-mercerized yarns has been completed. The data showed no apparent effect of yarn ply twist on fabric wearability. In other work, 144 experimental fabric structures woven from plied yarns (60/2 and 40/2, Pima S-2) were treated with a commercial delayed-cure resin and fabricated into pants cuffs for wearability testing. These tests are now in the final stages. Four cuffs made from each fabric have already undergone 45 launderings; 14 of the 144 structures have yet to show signs of wear. A similar series of experimental fabrics will be woven from singles yarns and evaluated. Pima S-2 cotton has been spun into 20/1 and 30/1 yarns for duplicating in singles yarn the plied-yarn seersucker fabric previously sent to the National Cotton Council of America for service testing. Based on favorable results from these service tests, the Council has initiated a test marketing program for the plied-yarn fabric tailored into men's seersucker suits. Other work in progress involves exploratory research to determine the effect of polymer-treated yarns on the abrasion resistance of fabrics woven therefrom. (S2 1-254).

Further improvements in the abrasion resistance of wash-wear and durably pressed sateen and corduroy fabrics have been achieved by applying a very low add-on of a face coating (polymer-forming, crosslinking resins) to the fabric prior to preferential crosslinking of the back side of the fabric. Sateen fabrics that were face coated with selected formulations and then cured, followed by back coating and curing after fabrication into pants cuffs, were washed and tumble dried 17 times before any evidence of abrasion was noted. This performance equaled that of a control cuff containing 15%

nylon/85% cotton. A corduroy fabric, face coated with a triazone resin and cured and then back coated with dimethylol dihydroxyethyleneurea, withstood 20 washing and tumble drying cycles with excellent retention of the pile, adequate retention of creases, and retention of good smooth-drying properties. Tests made to separately assess the abrasive damage of washing and tumble drying indicated that washing caused about 4 times as much abrasive damage as tumble drying. Tests made on pants cuffs fabricated from an 8-1/2 oz. sateen fabric indicated that wet crosslinking followed by backcoating and delayed curing reduced abrasion damage during repeated washing and tumble drying cycles. Studies are currently in progress to determine the influence of stretch properties on abrasion resistance and wear life. Stretch in combination with durable press features is desirable because of greater comfort and possibly better wearing qualities. Work is underway to determine the effect of compacting uncured resin-treated fabrics for warp stretch on the properties of the fabrics. Work on applying grafted polymers followed by crosslinking of cotton has also been conducted. Fabrics treated with a graft polymer (Cellosolve acrylate) and then treated with a crosslinking resin (Permafresh 183) had improved abrasion resistance. Cooperative research with several textile finishers in the application of preferential crosslinking treatments to corduroy, twill and sateen fabrics is in progress to develop optimum treatments. (S2 1-288).

Research findings by the contractor (Georgia Tech Research Institute) on the causes of seam pucker should enable the development of improved sewing thread and sewing techniques for wash-wear cotton fabrics to overcome or minimize the problem. Evaluation of sewing techniques has been completed on untreated, Belfast finished, and Permafresh finished printcloth with mercerized and soft, untreated, treated and cured, and treated and uncured sewing threads. Sewability tests showed major differences among the threads and treated and untreated fabrics. The mercerized and soft untreated threads were sewable; the mercerized, cured triazine treated threads were partially sewable; DMEU treated, both cured and uncured, and triazine uncured thread were not. By means of high-speed photography it has been found that one of the major factors in deformation of fabrics during sewing is the feed dog presser foot combination. Needle penetration appears to be a minor cause of deformation. Sewing in the warp and filling direction caused distortion waves which crossed the line of stitches and were then trapped and sewed in place where they appeared as puckers. Waves in the bias direction did not cross the lines of stitches, thus were not trapped and no puckering developed. In research to predetermine the sewability of a thread, a study of selected physical characteristics of the threads indicated an apparent correlation between abrasion resistance and work of rupture as an index of sewability. Data from this correlation led to a five-zone array into which all test threads fell as sewable, not sewable, or indeterminant. By means of a sonic fatigue device that subjected the test threads to some of the stresses which occur in actual sewing, it was determined that the highest probability of thread failure exists at the stitch interlock point. These results agree with earlier findings obtained by sewing with the experimental threads. A photoelectric scanning device that

records the amplitude and frequency of seam distortion was developed. It appears that this unit may find widespread application as a quality control device in commercial garment manufacture. (S2 1-228(C)).

4. Weather-Resistant Cotton Fabrics. Continued good progress has been made in the cooperative research on weather-resistant cotton fabrics with the Canvas Products Association International and the Foundation for Cotton Research and Education. The previously developed copper borate-zirconyl ammonium carbonate fungicides have been compared with several commercial copper-8 treatments for outdoor cotton fabrics. Outdoor weatherings of the treated fabrics over the same 21-month period of exposure showed that the copper-zirconium treatments gave significantly better protection than that provided by the commercial treatments, and at a fraction of the cost. Further study and evaluation of zirconium-metal salt fungicides has demonstrated that treatments based on these agents afford excellent outdoor protection not only to cotton fabrics but also to jute, burlap and other low-cellulosic fibers. This opens up many new fields for application and use of these fungicides. It was also established that copper and silver-zirconium fungicides have additive advantages in imparting weather and rot resistance. Of particular significance is the finding that zirconium itself is strongly algaecidal. Fabrics treated with basic zirconyl acetate or zirconia by heat insolubilization techniques have remained free of algae over a three-year weathering period. Several companies are investigating this newly discovered property of zirconium. In other research, carbon disulfide has been found to react with zirconyl ammonium carbonate to produce a cotton treatment that demonstrates marked water-repellency characteristics. These and other approaches to the use of zirconium compounds for improving cotton's weather resistance will be investigated. (S2 1-259).

Cooperative research has also continued on the development of multipurpose finishes for outdoor cotton fabrics. Cotton fabrics treated with a new colorless weather-resistant finish based on trimethylolmelamine and zirconium acetate and then coated with a pigmented vinyl coating are still retaining 100% of their original strength after 24 months of outdoor weathering in the warm, moist climate of New Orleans. This promising new finish was developed primarily to increase the service life of cotton fabrics used in coated products, and its lack of color makes it particularly desirable for white or light-colored coatings. Experimental weather- and mildew-resistant, fire-retardant finishes which are effective at less than 25% add-on have also been developed. A more extensive study of these finishes is in progress. Also, samples of Army duck treated at low levels of add-on (2, 3, and 4%) with mixtures of selected light-screening pigments and fungicidal pigments have now completed four years of outdoor exposure. Approximately one-third (18) of these samples retained 30-40% of their original strength and are free from biological stain or growth. This performance is better than that of any previously known experimental treatments of equivalent weight add-on, and should be of real interest to finishers of outdoor fabrics. Other research, cooperative with the Air Pollution Division of the U. S. Department of Health, Education, and Welfare, has established that air pollution can

definitely cause accelerated degradation of exposed cotton fabrics. Samples exposed in highly contaminated atmospheres degraded four to five times faster than those in less polluted sites in the same geographic area. The establishment of this relationship will aid in further research to improve outdoor cotton fabrics. (S2 1-256).

The contractor (Texas Woman's University) is presently conducting the final exposure testing of fabrics in the research to develop weather-resistant, water-repellent finishes for cotton. Fabrics treated with selected water repellents in combination with weather-resistant finishes are under exposure at Calexico, California, and Denton and Brownsville, Texas. Results will be evaluated when the exposures have been completed. (S2 1-200(C)(Rev.).

Contract research at Southern Research Institute seeks to develop weather-resistant cotton textiles with improved properties by interfacial and graft polymerization procedures. Cotton printcloth, drill, and duck have been grafted with acrylonitrile, styrene, methyl methacrylate, and 4-vinyl-pyridine by several grafting techniques. Physical testing of these products is nearing completion. Electron microscopy studies confirmed previous indications of the dependence of graft polymer location and uniformity on the method of graft initiation. Radiation initiation results in the most uniform distribution of polymer. In soil burial tests, styrene-grafted fabric showed the highest resistance to rotting, and samples grafted with 4-vinyl-pyridine showed very little rot resistance. No improvement in weather resistance of the fabrics was achieved by the graft polymerizations, as evidenced by the results of 4 months' exposure of the fabrics to outdoor weathering. After exposure in a Weather-Ometer for 1,000 hours, printcloth samples grafted with 15% acrylonitrile by the ferrous ion-peroxide method had greater breaking strength retention than samples grafted by the post-irradiation method of initiation. Preliminary probing experiments on deposition of polyamide type polymers by interfacial polymerization techniques have been carried out. This work will be expanded to polymers other than the polyamides. (S2 1-245(C)).

Contract research has been initiated at Fabric Research Laboratories to develop improved coated cotton fabrics with optimum strength-weight characteristics for outdoor uses. Specially woven fabrics to be used in the research are being procured. Optimum fabric structures and coating systems for various end uses will be determined. (S2 1-244(C)).

5. Soil-Resistant Cotton Textiles. Additional research has been conducted to develop durable oil- and water-repellent finishes for cotton through the use of fluorochemicals. Extensive evaluations of the following three types of finishes were carried out: the APO-perfluorooctanoic acid finish; the ethylenimine-ethyl perfluorooctanoate finish; and the THPC-1,1-dihydroperfluorooctylamine finish. The first of these possesses a combination of properties that may make it useful as a specialized finish where only semi-durable oil repellency is desired. Although the finish is not very durable to laundering it has some durability to drycleaning, and many of the physical

properties of the treated fabric compare favorably with those of untreated cotton. The other two finishes will be useful where a high degree of durable oil repellency but low water repellency is desired. The high initial oil repellency obtained with the ethylenimine-ethyl perfluorooctanoate finish is durable to drycleaning and, in some cases, to ten launderings. This finish requires low add-ons and no cure step in its application. Many of the physical properties of the treated fabric compare favorably with those of untreated cotton. The treated fabric soils more than untreated cotton but ease of soil removal appears to be comparable, although the treated fabric remains more soiled than untreated fabric. A study of curing conditions for the THPC-amine finish indicates that an add-on of 2-4% gives very high oil repellency, which is very durable to laundering and has good durability to drycleaning. Even lower add-ons of this finish may also give useful properties. (S2 1-250).

6. Flame-Resistant Cotton Textiles, Including Those With Multifunctional Properties. Emphasis in recent research on durable, inexpensive flame-retardant finishes for cotton fabrics has been on the development of nonyellowing, strength-retaining finishes. Encouraging preliminary results have been obtained with new finishes based on: (1) tetrakis(hydroxymethyl)-phosphonium acetate, bis(methoxymethyluron), and zinc nitrate; (2) tetrakis(hydroxymethyl)phosphonium hydroxide-urea, and methylolmelamine or methylated methylolmelamine; (3) THPC, urea, and tetrabromophthalic anhydride. Fabrics treated with these finishes did not yellow during processing; finish No. 1 showed no yellowing even after the chlorine bleaching and scorching test. A flame-retardant finish obtained by impregnation of cotton fabric with THPC-polyphosphoramide solution followed by an ammonia-cure spray treatment proved to be semidurable. Strength retention of the treated fabrics was high. Good flame resistance and wash-wear properties were imparted to cotton fabrics by pad-dry-cure treatment with APO-phosphoric acid solutions. Use of the phosphoric acid as catalyst results in higher efficiencies and better fabric properties than when a latent acid catalyst is employed. It appears that the APO-phosphoric acid treatment promotes more polymer formation and less crosslinking of the cellulose. Techniques for imparting flame resistance as well as improving abrasion resistance and strength properties are being investigated. Additional basic information on flame retardancy is also being obtained to aid in the development of improved flame-retardant finishes. Untreated cotton samples of different degrees of purity have been found to have different decomposition paths, as measured by differential thermal analysis (DTA) and thermogravimetric analysis (TGA). This indicates that DTA and TGA methods can be used to detect small amounts of noncellulosic materials in cotton. (S2 1-257).

Further research to impart multifunctional properties to cotton in a single treatment has led to the discovery of six new organophosphorus crosslinking agents, all of which impart a relatively high degree of wet wrinkle recovery and a modest amount of flame resistance. The new agents are: tris(chloromethyl)phosphine oxide; tris(bromomethyl)phosphine oxide; methyl bis(chloromethyl)phosphine oxide; methyl bis(iodomethyl)phosphine oxide;

bis(sulfatomethyl)phosphinic acid; and bis(iodomethyl)phosphinic acid. Improvements in dry wrinkle recovery and flame resistance can be obtained by subsequent treatment with nitrogenous crosslinking agents. Deposition of polymers in cotton to improve strength, abrasion resistance and other properties is also being investigated. A polyamide was successfully introduced into cotton by the internal interfacial polymerization of adipoyl chloride and 1,6-hexanediamine. Methyl methacrylate was polymerized in cotton fabric by both liquid phase and vapor phase techniques. The latter technique with ammonium persulfate catalyst was most successful. The various products are under evaluation. (S2 1-251)

7. Cotton Textiles With Improved Luster. Lustrous wash-wear cotton fabrics with increased strength and durability have been developed. These lustrous broadcloth fabrics (112 x 60) were produced by weaving two-ply (60/2) combed grey yarns that had been mercerized slack and restretched to their original length, or to 3% above normal length, and then crosslinking the unscoured or scoured fabrics with DMEU resin. The tensile and tear strengths of the crosslinked fabrics were equal to or greater than those of the uncrosslinked grey control fabrics. After 20 standard launderings the crosslinked fabrics retained their strength, wrinkle resistance, and luster; their overall strength (both breaking and tear) equalled or exceeded that of the unmercerized or mercerized uncrosslinked control fabrics. Thus, the strength losses previously associated with wash-wear finishing of cotton have been overcome by use of the special yarn mercerization techniques. Broadcloth fabrics made from grey and mercerized yarns will be crosslinked with various commercial wash-wear finishing agents in combination with chemicals added to improve flex abrasion resistance. (S2 1-267).

8. Stretch and Bulked Cotton Products. Completed contract research at Clemson University has indicated several useful techniques for achieving increased bulk, warmth, and dimensional stability for cotton knit fabrics. Of several approaches investigated, the technique of applying finishing agents such as DMEU or APO by vacuum impregnation, centrifuging, then drying and curing, proved best. Tumble dry-cure methods produced finished fabrics of optimum volume (bulk); tenter drying and curing, though not quite as good, would be more practical for commercial use. Treatment of other than jersey cloth by vacuum impregnation-centrifuging followed by tumble dry-cure was superior to soak-pad, loop dry-curing. Vapor phase treatments with formaldehyde-hydrogen chloride systems gave adequate but not superior results. Polyurethane formation within the fibers did not give significant improvements in bulk. (S2 1-205(C)).

In contract research by North Carolina State University on evaluation of stretch-type cotton yarns in knit wear, the short-term service wear tests on T-shirts and socks knitted from resin-treated stretch cotton yarns produced by the back-twist and false-twist methods, and from untreated cotton yarns as controls, have been completed. The results of these preliminary performance tests indicated that, in comparison with the control garments, the stretch garments had: (1) a greater tendency for damage during wearing

and washing, (2) a greater tendency toward dimensional growth due to wearing and washing, (3) a higher rate of decrease in bursting strength in subsequent wearings, and (4) lower ratings in some aesthetic characteristics. Larger scale service tests, which will give a better picture of the performance of the cotton stretch articles in actual use, are in progress. The stretch garments are being compared with the control garments in actual wearing and washing tests over an extended period of time. One hundred students are wearing the garments in these service performance tests. Results obtained in the tests will be correlated with the physical properties of the various fabrics. The information will provide industry with a sound basis for the possible production of these new stretch fabrics and garments. (S2 1-197(C)(Rev.).

North Carolina State University has also continued the contract research on production of stretchable knitted cotton wearing apparel by slack mercerization. Investigations of the effects of yarn size, yarn twist, and mercerizing conditions on the stretch and recovery properties of slack mercerized knitted fabrics have shown that: (1) fabrics made of medium to coarse yarns have maximum elongation but minimum recovery, whereas fabrics of fine yarns have minimum elongation but maximum recovery, (2) fabric elongation increases with increased yarn twist, but yarn twist exerts little influence on fabric recovery, (3) maximum elongation and recovery occur when fabrics are mercerized in approximately 17% caustic soda, and when mercerized and washed at lower temperatures. No appreciable difference in stretch or recovery was noted between fabrics knit with two ends of 16/1 yarns and those knit with one end of 8/1 yarns. Additional studies on lightweight T-shirt fabrics and stretch socks produced by slack mercerization techniques were made. It was found that the ease of stretch of slack mercerized fabrics increases slightly with increased length of yarn per knitted stitch or loop, but recovery from elongation decreases with increasing length of stitch. Loosely knitted fabrics made from yarns of conventional "Z" twist had the best recovery after stretching, but knitting with yarns of "S" and "Z" twist in alternating courses eliminated fabric skewness. Oversized T-shirts were fabricated from loosely knitted fabrics, the pattern used for cutting the T-shirts being approximately twice the length of a conventional T-shirt to compensate for shrinkage occurring during the subsequent slack mercerization. The slack mercerized T-shirts were distorted around the collars and along the seams where the sleeves were sewn onto the bodies; and the front and back of the shirts tended to cling to each other, which is another objectionable feature. The T-shirts lost much of their stretch after bleaching. Stretch socks of different constructions were produced which had a range of up to 3 sizes after slack mercerization; however, much of this stretch was also lost on bleaching. Attempts are being made to correct these deficiencies. (S2 1-224(C)).

Data recently obtained in in-house research should aid industry in producing improved woven stretch fabrics from cotton. Valuable information on the effects of different types of commercial dyes, resins, and softeners on the properties of slack mercerized, filling-stretch cotton fabrics (80 x 80

printcloth) has been developed. The type of dye used (vat, sulfur, and naphthol) did not have any significant effect on stretch properties or other physical properties. Use of Permafresh LCR or Aerotex Resin 23 for fabric treatment resulted in better recovery after stretching and better wrinkle recovery but caused lower abrasion resistance, breaking strength and tearing strength than when dimethylol ethyl carbamate was employed. Fabrics treated with Rhonite R-1 resin had properties that were intermediate. Most fabric properties were improved by applying a softener in the resin bath. Of the softeners evaluated, a polyethylene type applied at the 1% level resulted in best fabric properties. Slack mercerized, scoured, dyed and resin-treated plan and twill weave fabrics had much better breaking strength, tearing strength and flex abrasion resistance in both warp and filling than comparable fabrics which were not slack mercerized. A slack mercerized, resin-treated, compacted, cured and dyed fabric from a commercial mill was found to have good stretch and recovery properties as well as satisfactory dimensional stability and wash-wear properties. (S2 1-226).

Recent cooperative research with the National Cotton Batting Institute, the Textile Waste Association, the National Cottonseed Products Association, and the Foundation for Cotton Research and Education has been concerned with improving the production and the performance characteristics of chemically treated cotton batting, and aiding in its further commercialization. Many new resins and latexes have been evaluated for their efficacy in improving the performance of cotton batting. The chemical industry is also researching some new resins specifically designed for the batting application. New information on drying of the spray-damp in-process products--obtained in other cooperative work--has emphasized the importance of the control of humidity during the drying step. At least 12 companies are either producing the new chemically treated batting, Cotton Flote, or installing the process in their plants. Two major U. S. automobile manufacturers began using Cotton Flote in some models of their 1965 and 1966 cars, and a third auto manufacturer has tentatively approved the product as an alternate for other padding materials currently in use. One bedding company is now using Cotton Flote in high-grade mattresses. Firms in many foreign countries have also shown interest in the new process and product. Chemically treated cotton batting appears to have excellent potential for further growth in cushioning markets in the automotive, bedding, and furniture industries. (S2 1-269).

9. Insect-Resistant Cotton Bags. Encouraging progress has been made in research to develop cotton bags designed to protect flour, cornmeal, and other milled cereals from attack by insects during shipping and storage. This research is cooperative with the Stored-Product Insects Research and Development Laboratory at Savannah, Georgia, bag manufacturers, and the Textile Bag Manufacturers Association. A simple, inexpensive process involving calendering and treatment of cotton bag fabrics with a special insect-repellent formulation (an emulsion containing wax, piperonyl butoxide, and pyrethrin) appears quite promising for imparting insect resistance. Preliminary insect-exposure tests with small bags made from

cotton fabrics produced by this basic process gave such encouraging results that large-scale 18-month evaluation tests (75,000 pounds of flour and cornmeal in 100-pound bags) are now being undertaken. Several additives which retard the loss of piperonyl butoxide from treated fabrics have also been discovered. This enhances the possibility of extending the effective life of insect-repellent treated cotton bags. Further work in this area is underway. If good insect-resistant cotton bags can be developed, this should prevent the loss of cotton's export commodity bag market (about 70 million yards of sheeting annually) to multiwall paper bags and other competitive materials. (S2 1-271).

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AREA 2 - COTTONSEED UTILIZATION - FOOD

Problem. Cottonseed products, including approximately two billion pounds of oil derived from the annual domestic production of cottonseed, face increasing competition for markets. For its chief market, edible products, cottonseed oil must compete with other vegetable oils and animal fats. The nation's capacity for producing these oils and fats is so great that supplies can be expected to exceed both domestic and foreign demand for some time to come. Improvements in the quality and utility of cottonseed oil are needed to retain present and open new markets for the currently large and possibly greater future production.

Much research is urgently needed on the fungi and toxic fungal metabolites which may develop in cottonseed and its processed products. The mycotoxin problem is a potentially serious one for many agricultural commodities. Also, additional information is urgently needed on the chemical, physical, and biochemical properties of cyclopropene fatty acids in cottonseed and means of converting them into physiologically inert forms. A recent report states that cyclopropenoids are potentiating agents to aflatoxins in the production of hepatoma in trout. Research is urgently needed in these areas. Usually there is discrimination in the markets against 25% to 50% of the production of cottonseed oil due to the presence of reddish colors that are not removed by conventional commercial refining, bleaching and deodorizing methods. It is essential that information be developed on the chemistry of the pigments responsible for the off-colors, and that more efficient means be developed to eliminate them and thus upgrade the oils, particularly for use in margarine and shortening. Fundamental information is also needed on hydrogenation to permit production of improved modified fats and oils. Other areas in which markets for cottonseed oil need to be developed through research include fat emulsions for intravenous feeding and edible emulsifiers. Improved cocoa butterlike fats and other confectionery fats derived from cottonseed oil could also provide new markets for large quantities of oil, as could film-forming coatings for nuts, meats, and other feed products to prevent moisture transfer. An additional urgent need is the preparation of cottonseed flours or protein concentrates for human consumption in developing countries and to extend the supply of protein to meet the worldwide shortage. For further advances in utilizing cottonseed in food, information is needed in both the basic and applied areas of research.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, biochemists, chemical engineers, and microbiologists engaged in both basic and applied studies on cottonseed and its products to develop new or extended food uses for these materials.

Research to develop fundamental information on the chemical composition and

physical properties of cottonseed products is conducted at New Orleans, Louisiana, as a basis for efficient applied research to produce food products from cottonseed. Fundamental research on seed proteins and associated materials is being conducted by the Seed Protein Pioneering Laboratory. Other in-house research involves methods of correlating and predicting solubilities of homologous and analogous long-chain saturated and unsaturated fatty acid derivatives. A major effort involves the isolation of cyclopropenoids in cottonseed and cottonseed oil, and investigation of the chemical and physical properties of these compounds with regard to their effect on the preparation and use of cottonseed products, the Foundation for Cotton Research and Education contributes toward this research. Additional research on chemical composition and physical properties of cottonseed and its products in relation to food is carried out under several contracts: (1) fundamental investigations of chemical transformations of olefinic compounds of fats and other agricultural materials by hydroboration and subsequent reactions to develop basic information for the production of useful products, at Purdue Research Foundation, Lafayette, Indiana; (2) biological studies of cyclopropenoid derivatives and cottonseed oils treated to remove cyclopropenoids, at Ralston Purina Company, St. Louis, Missouri; and (3) isolation and identification of the reaction products of gossypol with simple esters and model triglycerides, at Savannah State College, Savannah, Georgia. Additional work in this area is conducted under a grant to Boston University, Boston, Massachusetts, on the development of procedures for synthesizing C^{14}-labeled malvalic acid esters.

Research in the area of microbiology and toxicology as they apply to food products is being conducted at New Orleans, Louisiana. It concerns the isolation, identification, evaluation, and control of fungi and toxic fungal metabolites that may develop during the processing of cottonseed and is designed to improve the acceptance of processed products made from this oilseed.

Technology for the development of new and improved processes and products is the objective of research that is also conducted at New Orleans, Louisiana. In one current project, processes for making cocoa butter-like fats and improving their performance are receiving attention. Another area concerns the development of new and improved techniques for preparing useful derivatives of cottonseed oils by esterification and interesterification. The Office of the Surgeon General is supporting research on fat emulsions for intravenous nutrition, which is conducted cooperatively with the U. S. Army Medical Research and Nutrition Laboratory and several medical school research groups. Other current work--supported by the Agency for International Development--involves a study of the preparation of cottonseed flours and their derived products for human consumption in developing countries. UNICEF cooperates by arranging nutritional evaluations of experimental products in developing countries, and the Human Nutrition Research Division, ARS, also cooperates by evaluating certain of the products. Informal cooperation is also maintained with industry in connection with the research on new and improved food products and processing technology.

Other research on chemical composition and physical properties is in progress under grants of P. L. 480 funds to the following foreign institutions: Kyoto University, Kyoto, Japan; for an investigation of the chemical composition and reactivity of the nucleic acids of cottonseed, to obtain basic information needed for the increased utilization of this commodity (project duration - 3 years); University of Rome, Rome, Italy, for basic investigations on the physical and physicochemical properties of cottonseed proteins (project duration - 5 years); Hebrew University, Hadassah Medical School, Jerusalem, Israel, for development of methods for the improved preparation of protein hydrolysates for the determination of amino acids, to provide a more accurate means for assessment of protein quality and nutritive value of oilseed proteins, thus contributing to their increased utilization (project duration - 3 years); University of Bombay, Bombay, India, for a study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes (project duration - 5 years); and for a study of the synthesis and properties of pure saturated diacid and triacid triglycerides for use as model compounds in obtaining basic information needed to improve the utilization of cottonseed oil (project duration - 5 years); and Commonwealth Scientific and Industrial Research Organization, Ryde, Australia, for an investigation of the chemistry and biological effects of cyclopropenoid compounds that occur in cottonseed and its products (project duration - 5 years).

Additional research in microbiology and toxicology is in progress under a grant of P. L. 480 funds to the following foreign institution: Nagoya University, Anjo, Aichi, Japan, for studies of the biochemical mode of action of aflatoxins and their biodegradation, to obtain basic information needed for control of these toxins in cottonseed and other agricultural commodities that may be exposed to contamination by Aspergillus flavus (project duration - 3 years).

The Federal in-house scientific effort devoted to research in this area totals 25.2 scientific man-years. Of this number, 12.6 are applied to chemical composition and physical properties, 5.0 to microbiology and toxicology, and 7.6 to technology--process and product development. The domestic contract and grant research involves an additional 3.3 scientific man-years, all of which is on chemical composition and physical properties. P. L. 480 research involves 7 grants, 6 of which are on chemical composition and physical properties, the remaining grant being on microbiology and toxicology.

The following lines of work were terminated during the year: (1) contract research on the investigations of gossypol esters and of mild oxidation products of gossypol and gossypol derivatives to develop information needed to aid the production of cottonseed oils and meals of highest quality; (2) studies on the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components, to obtain

fundamental information to develop improved edible products and thus to expand utilization of cottonseed (P. L. 480 project)(both under Chemical Composition and Physical Properties); and (3) development of methods for upgrading the quality of cottonseed oil by improving the color and eliminating undesirable components such as cyclopropene acids (under Color, Texture, and Other Quality Factors).

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 4.0 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. The compositions, properties, structural factors and reactions of oilseed proteins and associated materials are being investigated in a program of pioneering research conducted by the Seed Protein Pioneering Research Laboratory. The fundamental information developed should lead to new concepts and possibly new applications for oilseed proteins, including cottonseed proteins. Since peanuts were found to be an especially suitable experimental material and were employed for much of the early pioneering research on seed proteins, the report of progress in the research is given in Area 5, "Peanut Utilization - Food."

A fundamental investigation of the physical and physicochemical properties of pure isolated cottonseed proteins is being conducted under a P. L. 480 project at the Institute of Biological Chemistry, University of Rome, Italy. A monodisperse major protein component has been isolated from the protein extracted from a glandless variety of cottonseed. This protein, designated as Acalin A, has been characterized through amino acid analysis employing different methods of hydrolysis, end group analysis, dissociation into subunits of lower molecular weight, and solubility under different conditions. A second major protein named Acalin B by the investigators, has also been isolated and purified. The chemical structure of both of these proteins is under investigation. Several enzymes present in resting cottonseed have been separated and are being subjected to purification. Two purified enzymes, transiaminase and malic dehydrogenase, have been studied in comparison with enzymes from animal sources. Basic information of the type being developed is needed in the potential application of cottonseed proteins to human food needs. (UR-E15-(40)-33).

2. Chemical and Physical Properties of Cottonseed Pigments. Development of the basic chemistry of gossypol offers promise of applications to improve cottonseed oil color and cottonseed meal quality. This problem was investigated under a recently terminated contract project at the University of Tennessee. Through the use of ferric chloride in the

presence of acetic acid, gossypol was successfully oxidized to the corresponding 1, 4 binaphthyl quinone. Quercetin, a common flavanoid pigment present in cottonseed, was found to inhibit the oxidation of gossypol by ferric chloride to gossypolone when the molar ratio of iron to quercetin was 1:1. However, no inhibition was noted when an excess of ferric chloride was used. Gossypol was also oxidized by manganese dioxide, but the products of oxidation have not been characterized. An indication of the structure of the addition product of gossypol and ethanol was obtained by NMR spectroscopy. However, no encouraging results were obtained when the molecular oxidation of gossypol in the presence of an aliphatic amine and cupric ions was attempted. The information developed should be of value in research to determine the nature of the physiologically active derivatives formed in cottonseed meals and the role of iron in the metabolism of cottonseed meals. It may also open the way for improved methods for the determination of gossypol. (S4 1-103(C)).

3. Chemical, Physical, and Biochemical Properties of the Oil and Fatty Acids, Including Cyclopropene Fatty Acids. In cooperative work conducted with WU and LSU and supported by the Foundation for Cotton Research and Education, three simple processes have been developed for the inactivation of cyclopropenoids in refined and bleached cottonseed oils by heating them under vacuum in a pilot-plant deodorizer in the presence of cottonseed fatty acids. Other progress was achieved in the determination of procedures to isolate the cyclopropenoids. Reversed phase chromatography on relatively large chromatographic columns provided small amounts (several grams per batch) of quite pure malvalate and sterculate. Experiments conducted at both NU and SU indicate that counter double current distribution can be used to produce sizable amounts of purified cyclopropenoids, which are needed for biological and chemical testing. A method of analyzing for cyclopropenoid acids in cottonseed and other oils by hydrogenation and gas liquid chromatography has been devised. Malvalic and sterculic acids were the only cyclopropenoids found in cottonseed oil. The cyclopropanoids, dihydrosterculic acid and dihydromalvalic acid, were found to occur as glycerides in cottonseed oil. (S4 1-135).

Work has recently begun on a grant awarded to Boston University to develop a technique for synthesizing cyclopropene acids labeled at specific carbon atoms with carbon 14. Research will be directed toward the development of reliable methods for the synthesis of malvalate esters, 7-(2-n-octyl-1-cyclopropene)-heptanoates tagged with radioactive carbon at the methylene carbon of the cyclopropene ring as well as at positions 1, 7, 8, 9, or 10 in the molecule. The phase of the problem involving the cyclopropenoid moiety is presently being investigated and, after it is solved, the synthesis of C ^{14}-labeled esters of malvalate will be undertaken. (S4 1-134(Gr)).

In a P.L. 480 research project being conducted at the Division of Food Preservation, Commonwealth Scientific and Industrial Research Organization, Ryde, New South Wales, Australia, a study is being made of the chemistry and biological effects of cyclopropenoid fatty acids that occur in

cottonseed and cottonseed products. These fatty acids, malvalic and sterculic, occur in the seeds of many plants of the Malvales order and are known to cause adverse physiological responses when fed to several animal species. Methyl esters of these two acids have been isolated separately from Sterculia foetida seed oil and have been used in feeding studies with hens and in in vitro studies in which methyl sterculate was shown to inhibit the conversion of stearate to oleate. Efforts are continuing to increase the efficiency of the incorporation of C^{14} into cyclopropenoids by seed slices and to chemically synthesize sterculic and malvalic acids labeled with C^{14} in the ring methylene system for use in tracer studies. Information obtained through the use of these materials in chemical and biological tests will be of value in the extensive domestic research program to assess the significance of cyclopropenoids in food and feed uses of cottonseed production. (UR-01-(40)-2).

In P.L. 480 research at the British Food Manufacturing Industries Research Association, under a project completed during the past year, an investigation was conducted on the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components. The fatty acid composition of a number of oils from cottonseed of various origins and processing histories was examined by several different methods. Gas-liquid chromatography yielded results nearest to the accepted true values. Fractionation by low temperature crystallization indicated that, although cottonseed oil contains 3 major component fatty acids, only 4 out of 26 probable triglycerides occur to the extent of over 8%, and the minor component acids are very uniformly distributed throughout the glyceride components of the oil. Data obtained from lipase hydrolysis in experiments indicated a marked tendency for the 2-position in the triglyceride molecule to be occupied by an unsaturated acid, and some indication was obtained of overall selectivity of linoleic over oleic acid to occupy this position in cottonseed oil. This led to the development of the concept of "selectivity coefficient," a factor that is constant irrespective of the composition and mean unsaturation of the oil. This concept is not only of considerable biochemical interest, but is of practical importance, since it makes possible for the first time for the component glycerides of any cottonseed oil to be determined with respect to both amount and configuration merely from a knowledge of the component fatty acids of the oil. (UR-E29-(40)-26).

In P.L. 480 research at the University of Bombay, studies of the relationship of the substituent fatty acid groups to the physical properties of diacid triglycerides of certain saturated fatty acids, including those that occur normally in cottonseed oil are now nearing completion. The diacid triglycerides of interest in this work are those containing one or two molecules of palmitic or stearic acid, and two or one of even-carbon saturated fatty acids of the series from acetic to stearic acid. A number of such diacid triglycerides of both the symmetrical and unsymmetrical configuration have been prepared and purified to around 99.8% purity, as determined by the most sensitive available methods. Physical properties such as melting point (of the β form), density, molar volume, refractive index, molar refractivity,

and viscosity have been obtained for 40 pure glycerides. In the final stages of the project, properties of mixtures of these pure glycerides, taken two at a time, will be determined. The methods of synthesis and purification developed in the course of the project together with data obtained in it will be of fundamental value as the basis for the further development of fats and oils specifically tailored for special food and industrial end uses. (UR-A7-(40)-3).

A recent advance in the basic study of solubilities of long-chain fatty acid derivatives was the design and construction of an apparatus that permits solubility data at temperatures as low as -40° C. to be determined by use of this apparatus, low-temperature solubility data have been obtained for stearic, palmitic, and petroselaidic acids in methanol, acetone, toluene, and isopropyl ether; for elaidic acid in the latter two solvent systems; and for stearic, myristic, and cupric acids in N,N-dimethyl formamide and N,N-dimethyl acetamide. The correlation method shows good linear relationships for the systems in which palmitic acid is used as the reference acid. In many solvents, stearic acid tends to give separate solubility curves for two polymorphic forms, the C and probably the B form, over part of the temperature range. These forms have been identified by X-ray and infrared measurements. Since palmitic acid gives only one curve, it is a better reference substance than stearic acid for use in applying the correlation method. Far infrared spectra obtained by the technique of FMIR (frustrated multiple internal reflectance) for stearic, arachidic, palmitic, petroselinic, erucic, and vaccenic acids appear to show differences among the first three listed; such differences among these three homologous fatty acids may be useful for identification and characterization. In addition, the binary system of palmitic-stearic acids has been constructed. Also, a number of solubilities of cyclohexylamine salts of fatty acids have been determined. The new solubility apparatus developed under this project permits a great deal of important fundamental data to be obtained with only small amounts of material and relatively few measurements and provides information that is requisite to the development of new crystallization techniques. (S4 1-129).

The systematic study of hydroboration of olefinic compounds of fats is continuing under contract to Purdue Research Foundation. Additional model compounds such as 3-butenylamine, 3-butenyl methyl sulfide, 1-methoxy-3-hexene, 3-hexenyl acetate, 3-hexenyl trifluoracetate, 3-hexenyl trichloroacetate, ethyl 3-hexenoate, and 1-chloro-3-hexene were synthesized and reacted with diborane and/or disiamylborane (di-s-isoamylborane) to produce organoboranes that were oxidized and hydrolyzed with alkaline hydrogen peroxide to produce the respective hydroxy compounds. The treatment of unsaturated esters with excess diborane results in both addition to the carbon-carbon double bond and concurrent reduction of the ester group, but the use of theoretical amounts of diborane minimizes the reduction of the ester group. However, the use of disiamylborane is more selective since it circumvents the partial reduction of the ester group. It was possible to react the organoborane prepared from 3-butenyl methyl sulfide with

alkaline hydrogen peroxide without oxidizing the sulfide to the sulfoxide or sulfone. 1-Methoxy-3-hexene was synthesized and reacted with diborane followed by contrathermodynamic isomerization prior to oxidation and hydrolysis to produce hydroxy compounds. Present results clearly indicate that the boron atom migrates toward either end of the aliphatic chain, equilibrating the boron atom among all positions in the molecule. When the boron atom, however, reaches the carbon atom beta to the methoxyl group, an E1 (unimolecular β-elimination) reaction takes place with the cleavage of the ether linkage to produce the corresponding terminal olefin, which also undergoes hydroboration. Amines were synthesized by the hydroboration of olefins such as 1-octene followed by in situ amination with hydroxylamine-O-sulfonic acid. The amination reaction was not affected by acid or base catalysis, but it was adversely affected by polar solvents. Time and temperature have some effect upon the amination reaction, but the best yields were produced within one hour at 67° C. or within three hours at 25° C. The fundamental information derived in this contract should aid in synthesizing fatty acid derivatives that are potentially useful industrial products. (S4 1-112(C)).

B. Color, Texture, and Other Quality Factors

1. Improvement of Color and Quality of Cottonseed Oil. Recent research to upgrade the quality of cottonseed oil by improving the color and eliminating undesirable components has placed emphasis on the removal or inactivation of cyclopropenoids. Simple and practical methods, readily applicable in existing refineries, have been found very effective for inactivating the cyclopropene fatty acids (chiefly malvalic) of cottonseed oils. Inactivation was carried out in a 400-pound capacity iron pilot-plant deodorizer. The three procedures consisted of heating an appropriate commercial refined and bleached cottonseed oil in the presence of: (1) a small concentration of added cottonseed acids and a small steam sparge under slightly reduced pressure prior to high vacuum deodorization; (2) a small steam sparge under slightly reduced pressure to generate in situ a relatively small amount of cottonseed acids prior to high vacuum deodorization, and (3) a large steam sparge and high vacuum to generate in situ a relatively large amount of cottonseed acids under high vacuum deodorization conditions. Although the second process appears to be most efficient, all reduced the malvalic acid from 0.57% to 0.01% or less. Although no work has been done recently on an earlier phase of this research -- development of a process for bleaching off-colored cottonseed oils with activated alumina -- contact has been maintained with a major company relative to proposed additional pilot-plant work on the process. (S4 1-114).

C. Microbiology and Toxicology

1. Investigation of Fungi and Toxic Fungal Metabolites That May Occur in Cottonseed and Its Products. Considerable progress has been made in several areas of the research directed toward control of fungi and toxic fungal

metabolites in cottonseed and its products. The SURDD aqueous acetone procedure for determination of aflatoxins in cottonseed products has been modified by inclusion of a silica gel cleanup column to provide significantly cleaner extracts for thin layer chromatography (TLC) and for better application to many agricultural products. This improvement permits detection of low or trace amounts of aflatoxin, a fact that may be important in future work on detoxification. Aflatoxins separated by TLC on silica gel plates can be determined with a precision of 3 to 4% by use of recording densitometer equipped for fluorescence measurements. Relative response in the solid state was in the order $B_2 > G_2 > B_1 > G_1$ with ratios of 4.3:3.0:1.7:1. Relative intensity of fluorescence of aflatoxins in solution was found to vary with the solvent: in $CHCl_3$, the order is $G_2 > G_1 > B_1 > B_2$; in methanol, $G_2 > B_2 > G_1 > B_1$. This objective instrumental method places the assays on a much more secure basis and also permits greater precision. Pure crystalline aflatoxins B_1 and G_1 have been isolated from aflatoxin concentrates by column chromatography on silica gel followed by fractional crystallization. Pure aflatoxins B_2 and G_2 were prepared from hydrogenation of B_1 and G_1, respectively. These materials are being used to prepare quantitative and qualitative aflatoxin standards. Continued worldwide distribution of aflatoxin standards, by affording a uniform reference, has doubtless improved the reliability of reports of its occurrence in various agricultural products.

Completion of an experiment on aflatoxin elaboration in cottonseed indicated that both sterilized and living glanded and glandless seed can be utilized as substrates by A. flavus with production of high levels of aflatoxin B_1, B_2, G_1, and G_2. Gossypol pigments are not a barrier to either mold invasion or aflatoxin production. Hulls, lint, and linters are poorly utilized by A. flavus. Preliminary evaluation indicates that their production of aflatoxin on cottonseed and other substrates differs markedly with temperature. Under experimental conditions, the impermeable coat of "hard cottonseed" has partially protected the seed for 21 days against the invasion of A. flavus and the subsequent production of aflatoxin. However, the rate of development of this type of seed after maturation may limit its usefulness in a possible solution to contamination in cottonseed. There is little problem with the oil, since alkali refining completely removes any aflatoxins that may be present. (S4 1-116).

D. Technology -- Process and Product Development

1. New and Improved Cottonseed Oil Products. The relatively simple process for the preparation from cottonseed oil stearine of a good cocoa butter-like fat, more compatible with cocoa butter than are commercial hard butters, has aroused a great deal of industrial interest. Chocolate-type bars prepared from the new fat had excellent gloss, bloom resistance, and good getaway when tasted. Research is continuing, however, on determining and improving the properties of various formulations. In a study of samples of cocoa butter, cocoa butter-like fat, and their mixtures, only slight changes in the amount of X-ray diffraction were found between

marked and unmarked samples. No crystal types other than those present in pure cocoa butter and the cocoa butter-like fat were found in the mixtures, a demonstration that they behaved as true mixtures and not as different compounds. However, the proportion of a particular crystal species differed among cocoa butter, the cocoa butter-like fat, and the mixtures. Dilatometric behavior of mixtures of fat HP-23F and cocoa butter were investigated at 12.5% intervals of cocoa butter content. Successive 12.5% composition differences cause a gradual change in melting properties; mixtures containing 50-75% cocoa butter are slowest to convert to higher melting forms. Thus each composition would require different solidification and tempering procedures to obtain the highest melting form. The cocoa butter-like fat was separated into some 90 fractions, some of which were converted to methyl esters and analyzed by gas-liquid chromatography. Minor components, dihydromalvalate, dihydrosterculate, and an unidentified component were found in some of the fractions. Elaidic acid was found to some extent as the dielaidic monosaturated glyceride. Attempts to produce a cocoa butter-like fat with a higher ratio of monounsaturated glycerides to diunsaturated glycerides did not yield a product significantly better than previous ones, which are now being evaluated by at least one company. (S4 1-125).

Experiments were continued to develop new and improved techniques for preparing useful derivatives of cottonseed oils by esterification and interesterification. Diglycerides were esterified with fatty acids under conditions that minimized interesterification to develop the optimum procedure for preparing mixed triglycerides of predetermined fatty acid composition. The best temperature for esterification with a sulfonic acid catalyst was 180° C. The most effective catalysts tested were the sulfonic acids, the most active being benzenedisulfonic. Equimolar quantities of dilaurin and oleic acid were allowed to react at 180° C. in the presence of 2-napthalenesulfonic acid while vapors of heptane were passed through the reactants to rapidly remove the water of esterification. The reaction products were analyzed for contents of mono-, di-, and triglycerides and for free fatty acids after 10 min. and after 4 hr. After 4 hr., the reaction product contained 67% triglycerides, consisting of 64% laurate, 0.9% palmitate, and 35% oleate. The results indicated that the reaction proceeded as desired. When equimolar quantities of oleic acid and mixed diglycerides of caproic and caprylic acids were allowed to react under similar conditions, 57% triglycerides were obtained, comprising 66% caproate, 26% caprylate, and 7% oleate. Apparently caprioic acid and possibly fatty acids of still shorter chain length possess a tendency to interesterify which is much greater than that possessed by lauric and other longer chain fatty acids. This rapid interesterification of the diglycerides of short-chain fatty acids to the corresponding triglycerides when esterification with a long-chain acid is attempted may be of value in the preparation of diglycerides of long-chain fatty acids. A large batch of amylose palmitate has been prepared by the reaction of palmitoyl chloride with amylose dispersed in a solution of pyridine, p-dioxane, and toluene. However, when novel methods of preparing amylose palmitate

by esterification and interesterification were tried, no reaction occurred, despite attempts to provide an extensive surface area, which evidently will not by itself cause a reaction between insoluble amylose and palmitic acid under ordinary conditions of esterification and interesterification. Additional catalysts will be evaluated for the esterification of partial glycerides and the mechanism of catalyzed esterification will be investigated. (S4 1-128).

Three major contributions have resulted from chemical investigations of cyclopropenoids conducted by the University of Illinois under a recently completed contract. As previously reported, three of the major pigmented components of the Halphen-test reaction have been isolated in chromatographically pure form and two of these have been identified. This information should help elucidate possible physiological reactions that might occur upon ingesting products that contain cyclopropenoids. The second result involved experiments designed to synthesize cyclopropene derivatives from cis- and trans-2-phenyl-1-methylcyclopropanecarboxylic acids. The acid group was converted to an acid chloride; the acid chloride group was converted to an acid azide; the acid azide group was converted to an amine; the amine group was converted to a quaternary ammonium salt; and attempts were made to decompose the trans-2-phenyl-N,N,N,1-tetramethylcyclopropylammonium iodide to produce 2-phenyl-1-methylcyclopropene. Such synthesis of precursors to produce model cyclopropenoids for comparative studies should ultimately aid in selecting the best system of elimination or inactivation. The third finding, for which preliminary data were previously reported, is of practical importance, since it involves the inactivation of the cyclopropenoid moiety of sterculene or methyl sterculate. Different types of reagents and various conditions were used in these reactions. Acetic acid alone, even at reflux, reacts rather slowly with sterculene; but systems, such as acetic acid and perchloric acid, acetic acid and boron trifluoride, and silver nitrate and silica gel, effectively inactivate the cyclopropenoid moiety of sterculene, even at room temperature, within a few hours. These three systems also inactivate the cyclopropenoid moiety in methyl sterculate under mild conditions; however, a longer period of time and/or higher temperature are required to inactivate the cyclopropenoids in refined cottonseed oil. This discovery of systems that perform effectively at mild conditions has considerable practical significance. (S4 1-107(C)).

Work on the development of cottonseed oil emulsions for intravenous nutrition is being continued in research that is conducted cooperatively with the U. S. Army Medical Research and Nutrition Laboratory as well as several medical schools and is supported by the Office of the Surgeon General. Cottonseed oil processed by the usual methods was found to be unsatisfactory for use in intravenous emulsions, whereas removal of pigments and polar components produced an oil that was very satisfactory with respect to physiological response. Spectral data and analysis by TLC indicated that bleaching earth removed only pigments, and alumina removed polar components and remaining pigments. To confirm the necessity

of removing these substances, portions of a solvent-extracted oil were processed to yield (1) refined, deodorized oil; (2) refined, bleached, deodorized oil; and (3) adsorbent fractionated, deodorized oil. Two emulsions of each oil were prepared, one with nonoxidized phosphatides and the other with pure lecithin as the stabilizer. Only emulsions of adsorbent fractionated oil were satisfactory. Phosphatides are susceptible to chemical change on storage, probably oxidation. An investigation of the effect of oxidation of phosphatides on their physiological response was begun, but preliminary evidence indicates that fresh, nonoxidized, phosphatides apparently are satisfactory for use as the stabilizer for emulsions prepared with pigment-free cottonseed oil. Emulsions prepared from purified oil gave completely satisfactory results in hemodynamic screening tests in dogs. The fact that the preparations can be frozen and thawed without adversely affecting the size of the dispersed particles of oil should facilitate adoption of such emulsions for intravenous alimentation. Another encouraging aspect is that no difficulties were encountered in the handling of large quantities (85 liters) of a fat emulsion. Plans include development of a quantitative analytical procedure for determining phosphatide composition and methods for detecting chemical changes in phosphatides used for emulsification. (SU-0-0-2(SG), SU-0-0-4(SG)).

2. Cottonseed Flours and Derived Products for Human Consumption in Developing Countries. Progress is being made toward developing processes for producing low-cost, high-quality cottonseed flours for human consumption in developing countries. This research is supported by the Agency for International Development (AID). Optimum conditions were determined on a bench-scale for using aqueous acetone for maximum removal of gossypol, fatty acids, and aflatoxins, and minimal removal of neutral oil, nitrogen, and sugars, and then extracting the oil with hexane or mechanical pressing. High quality cottonseed meals have been prepared. The high quality, low-fat cottonseed flours prepared at SU and evaluated by HN using a batter process yielded good breads of satisfactory texture and volume. They were a distinct improvement over the yeast breads previously made, but had a slightly bitter taste and greenish color. The cause of these off-flavors in acetone:hexane:water (AHW)- or acetone-extracted meals and flours has been pinpointed as diacetone alcohol, a high-boiling, minor component of commercial acetone. Although it is quantitatively removable from commercial acetone, diacetone alcohol is formed *in situ* from acetone during extraction and meal desolventizing. An aid in solving the problem of off-flavor is the development of a quick detection procedure. Preliminary investigations of other solvents showed that methanol, ethanol, and isopropanol bound gossypol excessively; butanone resulted in a characteristic, somewhat undesirable flavor in the flour; benzene involves serious health hazards; but ethyl acetate yielded a meal practically bland in flavor and low in residual lipids and in free and total gossypol. Fortunately much of the processing information obtained during the development of the AHW process and its modification would be directly translatable to a process based on ethyl acetate as a solvent. (SU-0-0-3(AID)).

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AREA 3 - COTTONSEED UTILIZATION - FEED

Problem. Cottonseed meal, used chiefly as a protein supplement in feeding ruminant animals, faces serious competition from synthetic urea and other supplements. The quality and nutritive value of the meal must be improved and new outlets developed to make optimum use of the meal produced from domestic cottonseed each year. Improvement in the quality and nutritive value of cottonseed meal is needed so that it can better compete with other protein feed supplements. Additional information is needed on the physiologically active constituents of the meal responsible for egg abnormalities, swine mortalities, and growth abnormalities of young animals that limit cottonseed meal's usefulness in poultry and swine rations, and for the reported implication of cottonseed meal in the incidence of trout hepatoma, which has resulted in its elimination from use in fish feeds in certain areas. Processing methods must be devised for the commercial production of meals that can be fed to broilers, laying hens, and swine, safely and without restriction.

A most important problem is the need for information on fungi and toxic fungal metabolites that may develop in cottonseed and its processed products. An urgent requirement is a practical method for inactivating or removing these toxic materials from contaminated seed, so that it can be utilized in feedstuffs rather than being diverted to use in fertilizers, which of course have a much lower value.

Much progress has been made toward the development of cottonseed meals that are fully suitable for all feeding needs. Additional basic and applied research is still needed, however, to develop practical and economic means to produce fully satisfactory meals during the processing of cottonseed without sacrificing the quality of the oil.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, biochemists, microbiologists, and chemical engineers engaged in both basic and applied research to develop new or extended feed uses for cottonseed and its products.

Research to develop fundamental information on the chemical composition and physical properties of cottonseed products is conducted at New Orleans, Louisiana, as a foundation for efficient applied research pertaining to their use as feeds. Fundamental research on seed proteins and associated materials is being conducted by the Seed Protein Pioneering Laboratory. In other in-house research, chemical composition and characteristics of the protein systems of cottonseed are being investigated to serve as the basis for improving the nutritive value of cottonseed flour and meal.

Certain areas of microbiology and toxicology as they pertain to cottonseed made into feeds are being investigated at New Orleans, Louisiana. One phase

of this work is directed toward the inactivation or removal of aflatoxins from contaminated cottonseed and its products to permit their utilization in feeds. Another line of research is the isolation and identification of cottonseed constituents that cause mortalities among swine.

Research on technology for developing new and improved processes and products is also being conducted at New Orleans, Louisiana. Processing conditions to produce high-quality oils and meals from glandless cottonseed are being investigated. In addition, suitable processes and processing conditions are being developed to permit the inactivation of aflatoxin in cottonseed products by use of basic nitrogen compounds. In addition to the in-house work, contract research on technology is being conducted by IIT Research Institute, Chicago, Illinois, on development of practical processing methods for inactivation of cyclopropene groups, which decrease the value of cottonseed meal as a feed for laying hens.

Animal tests in connection with the overall research program are conducted through the cooperation of nutritionists in State Agricultural Experiment Stations at universities, in the Animal Husbandry Research Division, and in industry. The Pharmacology Laboratory at the Western Regional Research Laboratory, Albany, California, cooperates by conducting animal studies to determine the physiological and pharmacological effects of cyclopropene acids and toxic fungal metabolites. Cooperation is also maintained with the Crops Research Division, ARS, Market Quality Research Division, ARS, the Food and Drug Administration, the National Cottonseed Products Association, UNICEF, and members of the cottonseed industry.

Additional research on microbiology and toxicology is in progress under grants of P. L. 480 funds to the following foreign institutions: Instituto Farmacologico "Mario Negri," Milan, Italy, for a study of the mechanism of gossypol toxicity counteraction by L-lysine (project duration - 5 years); and Regional Cooperative for the Protection, the Development and the Practice of Fishing in Valle d'Aosta, Valle d'Aosta, Aosta, Italy, for experimental studies to elucidate the role of cottonseed meal in the induction of hepatoma in rainbow trout to obtain fundamental information concerning the suitability of cottonseed meal for use in rations for this species (project duration - 3 years).

The Federal in-house scientific effort devoted to research in this area totals 13.9 scientific man-years. Of this number, 6.7 are devoted to chemical composition and physical properties, 2.1 to microbiology and toxicology, and 5.1 to technology--process and product development. The domestic contract research involves an additional 1.3 man-years, which are expended on technology--process and product development. P. L. 480 research involves 2 grants, both of which are in microbiology and toxicology.

The only line of work to be terminated during the year was a study of rates of extraction of cottonseed with acetone-hexane-water solvent mixtures, the

nature and quantity of constituents in miscellas, and rheological properties of marcs, to develop information basic to the production of cottonseed meal and oil of the highest quality (under Technology--Process and Product Development).

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 3.0 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Fundamental Investigations of Cottonseed Proteins. The composition, properties, structural factors, and reactions of oilseed proteins and associated materials are being investigated in a program of basic research conducted by the Seed Protein Pioneering Research Laboratory. The information developed should lead to new concepts and possibly to new applications for oilseed proteins, including cottonseed protein. Since peanuts were found to be an especially suitable experimental material and were employed for much of the early pioneering research on seed proteins, the report of progress in this research is given in Area 5 - "Peanut Utilization - Food."

2. Basic Research to Improve the Nutritive Value of Cottonseed Meal for Poultry and Swine. In the continuing investigation of the protein systems of cottonseed, work has been completed on the initial chemical characterization of ten meals submitted to Ralston Purina for nutritive evaluation under a cooperative agreement. This series of meals was prepared from quiescent and hydrated glandless seed by batch extraction with selected polar and nonpolar solvents to evaluate the effect of solvent extraction on protein solubility and nutritive value. Hydration of the seed enabled the more polar solvents to remove more of the non-nitrogenous materials, including the phosphorus, than with the quiescent counterpart; however, the defatting properties of acetone in the presence of water were greatly limited. Hexane-ethanol extraction of the hydrated seed gave the highest nitrogen content but the lowest percent of water-soluble material of all preparations. Amino acid analyses demonstrated only small variation in glutamic and aspartic acids, serine, and lysine. No significant variation was noted in the ε-lysine content. This observation that hydration has a significant effect upon the ease of extraction of cottonseed and upon the extent and nature of the materials extracted may have practical significance in the development of an optimum solvent system for extracting prepared cottonseed meats.

Results from another phase of the research indicate that it may be possible to partially fractionate the proteins of cottonseed on the basis of solubility alone. It has now been demonstrated that the proteins of cottonseed can be solubilized in an alkaline medium and acid precipitated. More than 80% of the nitrogen of defatted glandless cottonseed can be solubilized at

pH 10. Maximum extraction of nitrogen and minimum solubilization of phosphorus was achieved with a 0.2 M sodium carbonate-sodium bicarbonate, pH 10. More than 50% of the nitrogen solubilized was precipitated at pH 5. The color of the lyophilized protein that was acid precipitated from the carbonate-bicarbonate buffer was lighter and the phosphorus content lower than that obtained from sodium hydroxide extraction at pH 10. Exploratory studies on the isolation, fractionation, and characterization were conducted on proteins extracted by sodium chloride. Essentially all the nitrogen extracted from glandless cottonseed was soluble in 0.05 M carbonate-bicarbonate buffer, pH 10. Only a minor portion of the nitrogen but the major portion of the phosphorus precipitated on dialysis of the salt extract against the pH 10 buffer. Ultracentrifugal analysis showed two major peaks ~ 2S and 6S and a minor component of slightly higher molecular weight. It was determined that the low molecular weight component consisted of a number (8-15) of proteins that are water soluble and constitute almost 40% of the meal nitrogen. The 6S component consisted of one major and a few minor components. The major protein (40% of total N) was obtained in similar proportion in a variety of ways in various degrees of purity. In addition to the importance of the finding that there is this single major protein component, the alkaline buffer system developed not only provides a valuable research tool but also has commercial potential. (S4 1-130).

B. Microbiology and Toxicology

1. Investigations of Undesirable Natural and Adventitious Materials That May Occur in Cottonseed Meal. Continued research on the isolation and identification of cottonseed constituents that cause mortalities among swine are being conducted in cooperation with the Pharmacology Laboratory at WU and the Animal Husbandry Research Division, ARS. Investigations to determine the cause of deaths of pigs included a determination of the component fatty acids in fats from liver, heart, brain, and adipose tissues of rats receiving a meal that was highly lethal to swine. Statistical differrences were observed between these data and those obtained with rats receiving a high quality cottonseed meal extracted with acetone-hexane-water azeotrope. However, since the proportion of the respective fatty acids in the fats from the respective organs were of the same order of magnitude, the data provided no clue to the reason the toxic meal killed pigs. While the fatty acid patterns obtained from rats receiving the two meals were very similar, there were conspicuous differences in concentrations of stearic and oleic acid, probably due to malvalic acid present in the residual oil in the toxic meal. The fatty acid patterns of the organ fats were very similar to those observed with rats on a noncottonseed meal ration. A determination of the composition of the azeotrope extract of hexane-defatted cottonseed meal will be carried out in seeking a lead in the determination of the cause of swine mortality. (S4 1-136).

Research is in its initial stages under a P. L. 480 project at the "Mario Negri" Pharmacological Institute, Milan, Italy, in which a study is being made of the mechanism by which L-lysine appears to counteract gossypol

toxicity. Early findings indicate that tissue proteins in experimental animals are extensively bound by gossypol. Insight into the mechanism of gossypol toxicity has been afforded by the observation that intravenous injection is more toxic than oral or intraperitoneal administration. The finding that L-lysine does not exert a protective action against gossypol toxicity, while disappointing, does indicate the need for other gossypol inactivating systems, or the complete removal of gossypol from meals intended for feeds in nonruminants. Basic information being obtained in this research eventually will be applied to improving cottonseed meals for unrestricted feeding to animal species. (UR-E15-(40)-35).

Experimental studies designed to elucidate the role of cottonseed meal in the induction of hepatoma in rainbow trout is getting underway under a P. L. 480 project at the Regional Board for the Protection, Development and Practice of Fishing in the Valley of Aosta, Aosta, Italy. Cottonseed meals prepared by three different processes are being included in rations fed to two strains of trout, one of which is susceptible, the other resistant to the development of hepatoma. Data obtained from the research are expected to be helpful in clarifying the question of the suitability of cottonseed meal as an ingredient in rations designed for the feeding of hatchery trout. (UR-E15-(40)-44).

Another phase of the research directed toward utilization of cottonseed in feeds is concerned with the inactivation or removal of aflatoxins. A study of the effects of temperature, time of heating, and moisture on the aflatoxin content of a cottonseed meal (144 ppb B_1) showed that temperatures of 100°C. or above decreased the content of aflatoxin but the time-temperature required is so high that the nutritive quality may be severely diminished. The addition of moisture enhances the decrease in aflatoxin content. At 100°C., the lowest value of 33 ppb B_1 was achieved at 20% moisture and 120-150 minutes heating. Use of alkalies appears to be effective in lowering the aflatoxin content of cottonseed meal: calcium hydroxide (5% of meal) effected a reduction from 144 ppb B_1 to 4 ppb (50% H_2O added, heated 1.5 hr. at 84°C.). Treatment of a cottonseed meal (140 ppb B_1) with steam (20 min., 15 psig, 121°C.) yielded a product having 40 ppb B_1. This meal was also ammoniated in sealed containers under various storage conditions of temperature, time, moisture, and NH_4OH concentrations. Most significant reduction in aflatoxin occurred after two days' storage at 93°C., 15% moisture in the meal, and NH_4OH concentration equivalent to about 2% NH_3 based on the meal weight. Lesser aflatoxin reductions occurred under milder storage conditions. The meals stored at 66-93°C. were quite dark. Cottonseed meal (140 ppb B_1) was adjusted to 15% moisture, treated with 1% methylamine and stored in sealed containers at elevated temperatures. After 24 hrs. at 38°C. there was only a slight reduction in aflatoxin content, but after 3 hrs. at 93°C. aflatoxin was reduced 75%. Aqueous acetone (5 to 10% water) extraction of cottonseed meal (140 ppb B_1) reduced the aflatoxin 80-85%.

However, it is more difficult to inactivate aflatoxins in flaked cottonseed

meats, which contain oil, than in relatively oil-free cottonseed meal. Oily cottonseed flakes containing 970 ppb B_1 were heated with alkalies at 100°C. for 2 hrs. Of the four alkalies tried, only sodium hydroxide effectively lowered the aflatoxin to 36 ppb B_1. However, oil damage was indicated by the semisolid nature of the oil extracted from the product. Treatment of these flakes with 1.25% methylamine yielded products containing 0 to 5 ppb B_1, while ethanolamine yielded a product having 20 ppb B_1. Trimethylamine and choline were less effective. All of these amines were more effective in reducing aflatoxin content of cottonseed flakes than of contaminated peanut and cottonseed meals.

For use in cattle feeding experiments conducted at the University of California at Davis and monitored by WU, 6,000 pounds of cottonseed meal containing approximately 11,000 ppb B_1 were prepared at SU. Most of the aflatoxins used in these meals were prepared by NU. (S4 1-133).

C. Technology--Process and Product Development

1. Processing Technology Directed Toward Improved Cottonseed Products. In contract research conducted by the Illinois Institute of Technology Research Institute, chemical inactivation of residual cyclopropene fatty acids (CPA) in cottonseed meals, by means of sulfur dioxide, methyl mercaptan, or mixed cottonseed fatty acids has given promising results in laboratory-scale experiments. Optimum treatment conditions are presently being determined. Treatment of cottonseed meals with gaseous hydrogen chloride and carbon disulfide and with aqueous solutions of orthophosphoric acid and lactic acid were not successful in inactivating residual CPA. In another facet of the work, simple percolation with an acetone:hexane:water mixture yielded graded levels of residual CPA (3, 5, 8, and 29 ppm) in a commercial solvent - extracted meal and a screw-press meal that had originally contained 70 ppm and 170 ppm CPA respectively. The extracted meals and the unextracted controls were incorporated at a 20% level in the diets of laying hens. Evaluation of egg quality after 3 months' cold storage is in progress. The experiment should give useful data on the tolerance level of residual CPA in meals and on the synergistic effect of gossypol and CPA. This research may indicate that a certain level of residual CPA may be safely tolerated in rations for laying hens. If it is not necessary to completely remove or inactivate this component, the treatment of commercial meals will be greatly simplified. (S4 1-117(C)).

Another facet of the research to eliminate the undesirable physiological effects that may be associated with consumption of cottonseed is directed toward the use of glandless cottonseed. The processing conditions to produce high-quality oils and meals from glandless cottonseed are being studied in cooperation with the National Cottonseed Products Association; the Human Nutrition Research Division, ARS; and UNICEF. Acetone-hexane-water is being used for pilot-plant extraction to ensure removal of aflatoxins. Defatted meal was ground to flour, sifted, and sterilized at 180°F. The flour (920 pounds) was shipped to Guatemala for experimental use in animal

feed and in human food formulations. Chemical analysis was: lipids, 1.2%; total gossypol, 0.009%; protein, 60.1%; and lysine, 4.03 g/16g nitrogen. Preliminary comparison runs--one with glandless (1963 crop), one with glanded seed--have been made on the bench-scale cooker-desolventization assembly that was recently installed. Conditioning temperature of flakes and other conditions were varied. Glandless and glanded cottonseed processed under identical conditions of preparation, extraction, and desolventization were closely comparable and showed about equal oil extractability. Results of analyses indicate that mild heat treatment (up to 180°F.) of either glandless or glanded flakes cause little if any decrease of epsilon-amino-free (EAF) lysine content. Heating to 212°F. definitely lowers the EAF lysine of glanded cottonseed but has no significant effect on glandless. Addition of alkaline solution spray during cooking caused some lysine reduction in the case of glanded cottonseed but had no appreciable effect with the glandless. The higher lysine values can be attributed to the absence of gossypol, which, in the case of glanded seed, apparently combines chemically with the lysine to reduce its availability. The improvement in crude oil color by alkaline addition is more noticeable in the case of glanded seed than glandless. The oil samples produced in these runs are being evaluated by refining and bleaching tests. Data was also obtained on percolation rates of solvent through the cake. This bench-scale work indicates that glandless cottonseed can be efficiently processed by commercial plants presently employing direct hexane extraction with little or no modifications in either process or equipment; both oil and meal should be of superior quality. Thus glandless cottonseed offers an outstanding opportunity to the cottonseed industry to improve the quality and competitive position of its products and to expand and strengthen their markets. (S4 1-127).

Demonstration that removal of oil from cottonseed flakes by means of the acetone-hexane-water azeotrope is extremely rapid (60-90 seconds) and that thereafter the extraction is a simple washing extraction, coupled with successful application of a centrifuge to separate marc and miscella, affords the basis for a new and economical process for converting oilseeds to high quality oil and meal at lower cost than processes currently in use. The concentration of oil in the entrained miscellas in the several steps of the countercurrent extraction follows the universal dilution law, and the quantity of entrained miscella is an exponential function of the centrifugal force used in separating marc and miscella. Optically void miscellas are obtained. The method can be used without modification for cottonseed, soybeans, or a mixture of cottonseed and soybeans. Indications are that the liberation of gossypol from gossypol complexes in raw cottonseed flakes occurs on mild (carbonate) treatment of the flakes for a few seconds, and the treatment materially reduces the total gossypol in meals produced on the extraction of the raw flakes with the azeotrope. The gossypol contents of meals produced from glanded cottonseed are reduced in a stepwise countercurrent extraction to levels of less than 0.2% in periods of time of less than 5 min. when the kernels are comminuted in the presence of the acetone: hexane:water azeotrope. Gossypol and oil contents of the marcs of

comminuted cottonseed kernels and oil of comminuted soybean kernels follow a well-defined law (log L = a - RbP) in a stepwise countercurrent extraction when either the centrifuge or the vibrating screen is used to separate marc and miscella. L is the oil (or gossypol) content of the marc produced at pass P, a is log oil (or gossypol) content of the raw kernels, b is log of the ratio of the oil (or gossypol) content of a marc to that of the next succeeding marc (and is dependent only on the solvent-to-kernel ratio), R is a constant determined by the quantity of miscella entrained in the marc and is dependent upon the centrifugal force or the extent of drainage on the screen. Extraction with the azeotrope is very fast and the extent of extraction is described by the above law. Values of R, b, and P are independently selected and the operator may, through his selection, produce a meal of predetermined L. Development of this simple mathematical expression relating oil (or gossypol) content of an oilseed meal with processing variables should greatly facilitate oil mill operation. (S4 1-123).

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AREA 4 - COTTONSEED UTILIZATION - INDUSTRIAL PRODUCTS

Problem. Increased markets are needed for cottonseed oil, which is now produced in surplus. Such markets can be developed through research and development work leading to the production of entirely new and valuable industrially useful products and chemicals from this oil. Research on industrial uses should be expanded to develop new large volume outlets, such as markets in coatings, plastics, plasticizers, lubricants, surface active agents, elastomers, pesticides, and other chemical agents. The preparation of synthetic organic chemicals consumes large quantities of raw materials and intermediates, billions of pounds per year, and represents one of the fastest growing chemical industries. Application of new reactions and techniques for chemically modifying fatty acids derived from cottonseed offers promise of expanding industrial markets. Research is needed to develop procedures for carrying out such reactions, to characterize the products obtained, and to evaluate their utility in a wide variety of industrial products.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, and analytical chemists engaged in both basic and applied research to develop new industrial uses for cottonseed and its products.

Research to develop new and improved industrial products and processing technology is conducted at New Orleans, Louisiana. Present emphasis is on the preparation of amide derivatives of long-chain fatty acids and their evaluation as plasticizers, nitrile rubber softeners, lubricants, and antifungal agents. Informal cooperation is maintained with industrial firms for the evaluation of promising research products for specific end uses.

Research on chemical composition, physical properties, and structure is carried out under grants of P. L. 480 funds to the following universities: National Chemical Laboratory, Poona, India, for investigation of the synthesis and properties of new-type glycol mono alkyl ethers for control of water evaporation to extend the industrial utilization of cottonseed oil (project duration - 5 years); the Israel Institute of Technology, Haifa, Israel, for an investigation of π-complexed organometallic compounds derived from polyunsaturated fatty acids, to obtain fundamental information needed in expanding the utilization of cottonseed oil (project duration - 5 years); the Indian Institute of Science, Bangalore, India, for studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil to provide possible new outlets for utilization of the oil (project duration - 5 years); The Hebrew University of Jerusalem, Jerusalem, Israel, for a study of the preparation of new chemical derivatives from acrylonitrile and unsaturated fatty acids derived from cottonseed oil and other vegetable

oils (project duration - 4 years); and the Hebrew University Faculty of Science, Jerusalem, Israel, for an investigation of metalation reactions for the modification of mono- and dienoic fatty acids to provide increased functionability, thereby leading to possible new industrial applications for cottonseed and other vegetable oils (project duration - 5 years).

The Federal in-house scientific effort devoted to research in this area totals 2.7 scientific man-years, all of which is applied to technology--process and product development. P. L. 480 research involves 5 grants, all on chemical composition, physical properties, and structure.

The following lines of work were terminated during the year: preparation, characterization, and evaluation of derivatives from gossypol from cottonseed for use as biologically active materials, ultraviolet adsorbers, and other valuable products (P. L. 480 project)(under Chemical Composition, Physical Properties, and Structure); and contract research on ethylene copolymerization with unsaturated fatty acid compounds and selected derivatives of agricultural materials to extend their industrial utilization in commercial plastics (under Technology--Process and Product Development).

PROGRAM OF STATE EXPERIMENT STATIONS

The state stations did not report work in this area.

PROGRESS - USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition, Physical Properties and Structure

1. Fundamental Investigations Directed toward the Development of Industrial Products. In a recently completed P. L. 480 project at the University of Montevideo, in Uruguay, a fundamental investigation was conducted on the chemistry of gossypol and its derivatives having potential industrial application or utility. Gossypol, the yellow primary pigment of cottonseed, can be made readily available by isolation from byproducts of cottonseed oil refining. Its potential availability in quantity together with its reactive nature due to the several functional groups on its binaphthalene skeleton renders it a potentially attractive starting material for the synthesis of derivatives. Under this project a large number of derivatives of gossypol, many of which have not been heretofore reported, have been prepared and characterized. Among these are 24 new imino derivatives, 8 new ester derivatives, along with cyanohydrin and hydantoin compounds. Derivatives have been screened for potential usefulness as fungicides, germacides, ultraviolet screening materials and as potential anticancer agents. The information gained in this project may indicate promising industrial or other uses for this chemically reactive natural pigment. (UR-S9-(40)-2).

Work is progressing under a P. L. 480 project at the National Chemical Laboratory, Poona, India, in which an investigation is being conducted of

the synthesis and properties of new-type glycol monoalkyl ethers derived from cottonseed oil for the control of water evaporation. In early stages of the investigation much attention has been directed toward hydrogenolysis for the production of mixtures of cetyl and stearyl alcohol, and the preliminary evaluation of these as sources of surface layers to lower evaporation of the water substrate. Effort will now be devoted to reactions of the alcohols with ethylene oxide and to studies of the evaporation reducing effect of monolayers of the resulting ethers. Fundamental data obtained in these studies may be useful in the industrial production of materials from cottonseed oil having application in reducing evaporation from reservoirs in dryer regions of the U. S. (UR-A7-(40)-28).

An investigation of π-complexed organometallic compounds derived from polyunsaturated fatty acids in cottonseed oil is being conducted under a P. L. 480 project at the Israel Institute of Technology, Haifa, Israel. Early work under this grant has been concerned with the reaction of iron carbonyls with β-ionone and analogous conjugated dienes and trienes as model compounds. Attention has been directed toward the stereochemical problems and to the geometrical and positional isomerization of double bonds in these reactions. Based on these findings the research is now moving into areas concerned with the dienoic acids present in cottonseed oil. Fundamental information of the type being obtained in this work may eventually be applied in producing industrially useful materials from cottonseed oil derivatives. (UR-A10-(40)-34).

B. Technology--Process and Product Development

1. Research to Develop New Reactions and Products Suitable for Industrial Use. Nineteen additional N,N-dialkyl (symmetrical and unsymmetrical) fatty amides and one ester amide were prepared, characterized, and evaluated as polyvinyl chloride (PVC) plasticizers. Only three of these materials did not pass the storage compatibility test. These and previous data show that the total carbon count of the N-alkyl groups rather than that of the longest alkyl group is the compatibility-controlling factor. The plasticizing characteristics of the symmetrical and unsymmetrical dialkyl amides of the same total carbon count do not differ significantly; any N,N-dialkyl or alkyl-cycloparaffinic oleamide containing not more than 13-14 carbon atoms in the combined "alkyls" is compatible with PVC. Those amides having an N-cycloparaffinic substituent exhibit much poorer low-temperature performance, increasingly so with increasing ring size. Results of evaluation of 10 amides of erucic and Crambe acids submitted by NU were in accord with data for erucic and similar fatty acid mixtures previously published by SU. Antistatic tests show that many of the amides tested have some antistatic capabilities as coatings and some, those not containing a branched chain or cyclic group, retain this property even when incorporated as a component of the plastic composition. The superior plasticizer properties of the erucic over the oleic acid dimethyl amide suggests the investigation of the chemically modified C_{18} dimethyl amides of higher molecular weight; since the price of the dimethyl amine has recently been reduced from 28 to 18 cents

per pound, it is particularly attractive. Preliminary tests indicate that some of these amides may have utility as fabric softeners, and cooperative tests by EU do not rule out their use as lubricants. (S4 1-124).

Research is being continued by the U. S. Industrial Chemical Corporation to extend the industrial utilization of agricultural products in commercial plastics; the contractor is investigating the copolymerization of ethylene with unsaturated fatty acids and other selected monomers. Polymerization runs were made with vinyl laurate and erucylamide to complete the screening phase of the contract involving copolymerization of ethylene with 16 agricultural monomers. The runs with vinyl laurate proceeded smoothly, but the runs with erucylamide were more difficult, since it had to be kept hot to keep it in solution, and in a later attempt difficulties were encountered in pumping the erucylamide solution. A 50% solution in toluene was pumped at about 170°F., requiring the use of a different pump, which limited the comonomer concentration to a maximum of 3.8%. Virtually all the monomers had adverse effects on catalyst activity, an indication that future runs should be made at higher pressures. Low molecular weights of the products have limited the amount of testing that could be done by polymer test methods. Combined monomer content was determined on the products by physical separation and tests. In a second phase of the project, a large number of successful runs were made to copolymerize ethylene with selected fatty acid compounds. Copolymers of ethylene were prepared with vinyl laurate, methyl undecenoate, methyl esters of safflower acids, methyl esters of castor oil acids, methyl ester of dehydrated castor oil acids, methyl oleate, and methyl esters of linseed oil acids, under conditions designed to give the most useful types of polymers. Although it is difficult to predict the utility of the copolymers that have been prepared, some of the agricultural monomers may contribute slip agent properties and other products may have drying properties. (S4 1-115(C)).

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RELATED PUBLICATIONS OF STATE EXPERIMENT STATIONS

None

AREA 5 - PEANUT UTILIZATION - FOOD

Problem. Peanuts constitute a major cash crop in the Southern States and are in surplus. Because of their high price, domestically produced peanuts are used primarily (currently about 63 percent of the crop) in foods such as peanut butter, confections, bakery goods, and roasted and salted nuts. A critical problem in the utilization of peanuts, which has recently been made more clearly evident, is the sporadic contamination of peanuts by toxin-producing strains of common molds. The possibility of toxins entering foods intended for human consumption, as well as feedstuffs, is of the utmost concern. Intensified research is therefore urgently needed on the isolation, identification, evaluation, control, and inactivation or removal of mold toxins, such as aflatoxin, which may develop in peanuts and processed peanut products. New-type food products and improvement in the quality and uniformity of existing products are needed to increase consumer acceptance and extend markets for peanuts; the average per capita consumption has been rather stable since World War II. The increased trend toward mechanical harvesting has necessitated the use of artificial means for curing and drying peanuts, with the result that processed peanuts and peanut products do not always possess the same desirable flavor and physical properties as peanuts which have been cured slowly in the field. Information is needed as to the physical and chemical characteristics of those chemical constituents in peanuts which affect flavor, aroma, and other important properties of the processed products, as a basis for developing new or improved products and processing procedures. Fundamental studies of peanut proteins and associated materials could similarly lead to the development of new concepts and new uses.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, biochemists, analytical chemists, microbiologists, and chemical engineers engaged in both basic and applied studies on peanuts and peanut products to increase consumer acceptance and extend markets for peanuts.

Research to develop basic information on the chemical composition and properties of peanuts, its constituents, and processed peanut products is carried out at New Orleans, Louisiana. As a part of the Seed Protein Pioneering Research Laboratory's research on various seed proteins, fundamental investigations of peanut proteins and associated materials are conducted to form the basis for developing new concepts and perhaps new uses for peanuts and peanut proteins.

Research on the flavor of peanuts and their processed products is also conducted at New Orleans, Louisiana. One phase of this research includes investigations of the lipid or lipid-soluble constituents of peanuts and processed peanut products involved in the genesis of peanut flavor and

aroma. The Crops Research Division of ARS, the Consumer and Marketing Service, and several State Experiment Stations cooperate in the research by providing samples of peanuts of known variety and history. Additional research on flavor is being carried out under contract at the Agricultural Experiment Station, Oklahoma State University, Stillwater, Oklahoma, on a study of the relation of the carbohydrate, amino acid and protein components of the peanut to the formation of flavor and aroma during roasting.

Certain aspects of microbiology and toxicology as they relate to peanuts and their processed products are being investigated at New Orleans, Louisiana. An important line of such research is the isolation, identification, evaluation and control of fungi and toxic fungal metabolites, particularly aflatoxins, that may develop in these products.Related research is concerned with the development of economically feasible methods for the inactivation or removal of aflatoxins from contaminated peanuts and peanut products to permit their utilization in foods (and feeds). Cooperation is maintained with the Crops Research Division, ARS, Market Quality Research Division, ARS, State Experiment Stations, the Pharmacology Laboratory of WU, the Food and Drug Administration, industry, and nutritionists in USDA, at universities and elsewhere, in connection with this research. The problem of mycotoxins is also receiving attention in contract research at the Agricultural Experiment Station, Auburn University, Auburn, Alabama, on a study of the limiting environmental conditions for the elaboration of mycotoxins in peanuts; and at the Agricultural Experiment Station, Texas A&M University, College Station, Texas, to develop information relating processing methods, preprocessing history, distribution of immature, mature and germinating peanuts, and external conditions such as mold incidence as they affect consumer-use properties of processed peanut products.

Research on technology for the development of new and improved processes and products is being conducted at New Orleans, Louisiana. One project is concerned with the development of low-fat peanuts having acceptable peanut flavor and texture characteristics. Informal cooperation is maintained with peanut suppliers and processors, and with nutritionists and home economists for evaluation of experimental products as required. Other research, supported by the Agency for International Development, involves a study of the preparation of peanut flours and their derived products for human consumption in developing countries. Cooperation is maintained with UNICEF for arranging nutritional evaluations of experimental products in developing countries, and with the Human Nutrition Research Division, ARS, for evaluating certain of the products. Additional research on process and product development is being carried out under contract at the Agricultural Experiment Station, Auburn University, Auburn, Alabama, on the development of peanut products for use in preparation and fortification of processed and convenience foods; and at the Agricultural Experiment Station, Oklahoma State University, Stillwater, Oklahoma, on a study of sterilizing or inactivating treatments in conjunction with artificial drying and curing of peanuts to develop processing conditions needed for producing mycotoxin-free roasted peanut products of optimum quality.

Other research on chemical composition and physical properties is in progress under a grant of P. L. 480 funds to the following foreign institution: The University of Granada, Granada, Spain, for an investigation of the rate of reaction of protein with carbohydrates in peanuts, to provide information leading to improved peanut products, thereby increasing the utilization of this commodity (project duration - 3 years).

The Federal in-house scientific research effort in this area totals 11.6 scientific man-years. Of this number, 2.8 are devoted to chemical composition and physical properties, 1.4 to flavor, 3.3 to microbiology and toxicology, 4.1 to technology--process and product development. The contract research involves an additional 3.3 scientific man-years, 1.0 on flavor, 1.2 on microbiology and toxicology, and 1.1 on technology--process and product development. P. L. 480 research involves one grant on chemical composition and physical properties.

The only line of work to be terminated during the year was contract research on the isolation, identification, and characterization of flavor and aroma components of processed peanut products (under Flavor).

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 8.0 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. In pioneering research conducted in the Seed Protein Pioneering Research Laboratory, the ultrastructure of seeds, including peanuts, methods of enzyme and protein research, and protein synthesis in seeds are being investigated.

For several years a program has been underway here to develop techniques for staining seeds for electron microscopy in nonaqueous media. The reasons for omitting aqueous fixation was that such might create artifacts similar to those which arise at the onset of germination and would not enable an interpretation to be made of what exactly is happening in the resting seed. In previous years, there were reported descriptions of ultrastructure based on fixation with permanganate. The program has now been completed by development of techniques of osmium fixation as applied to dry cottonseed. Such a procedure has enriched the amount of detail made visible by the electron microscope. For example, it shows that the hyaloplasm contains numerous microtubules. These structures are still the subject of speculation as to their function but it is generally thought that they have a transport function in the cell. By this staining technique it has also been possible to see ribosomes in resting cottonseed.

Aleurone grains, the repository of the storage protein in seeds, have been isolated from peanut cotyledons by ultrasonic treatment of homogenates. That these were pure was demonstrated by observation with the electron microscope. No cytoplasmic protein was present; arachin was predominant. The total proteins of the embryo of the peanut contained mostly alpha-conarachin and very little arachin. This might suggest that there are two types of aleurone particles present in the peanut: one of which predominates in the cotyledon and another one in the embryo.

Electron microscopy of oilseeds shows structures which appear to be similar to those identified in the literature as spherosomes. The spherosomes of germinating peanuts were isolated and purified and shown to have the same structure in the isolated form as shown in intact tissue by electron microscopy. The spherosomes contain about 98% triglycerides and account for all of the lipid in the cotyledon. It can be calculated that the content of protein and phospholipid is just sufficient to account for a membrane surrounding the spherosomes. The spherosomes do not contain certain enzymes which are associated with fat metabolism. More about this is reported in a later section.

Globoids can be seen in ultrastructure as nonstainable spherical sections within the aleurone grains. These globoids have now been isolated from cottonseed homogenates. They contain 14% phosphorus, 1% of which is inorganic phosphorus and phosphorus-bound lipid; the remainder is phytin phosphorus. Globoids contain small amounts of protein which has a different amino acid composition than the protein of the entire aleurone grains. On the basis of the composition of phytic acid, these globoids can be said to contain 80% phytic acid.

Some of the properties of castor lipase were described in previous reports. These include the facts that it is present in the resting castor bean, is particulate-bound, and contains a lipid cofactor. There now can be added the finding that a soluble, low-molecular weight peptide can be removed from the lipase simply by incubation at acid pH. This removal is reversible; considerable progress has been made in purifying the peptide activator. Since the activator has a low-molecular weight, an examination was made of the ability of castor allergens to activate the apolipase. Crude castor allergens do indeed activate the apolipase. Work is continuing to determine whether there is any relationship between allergens and this peptide portion of the castor lipase enzyme.

The presence of an ATP phosphoinositol phosphotransferase, was demonstrated in the germinating peanut. This enzyme catalyzes the phosphorylation of ADP forming ATP and utilizes phytate as the phosphoryl donor. There is, however, nucleotidediphosphate transferase (adenylate kinase) activity in the peanut tissue. Hence, it is not clear how much of the formation of ATP is due to the phytic acid acting as a phosphagen and how much is due to the kinase. A rapid paper electrophoresis method of separating and identifying each of the phosphate esters of inositol has been developed.

The entire question of the possibility of phytic acid acting as a phosphagen was investigated from another viewpoint by the use of calorimetry to determine the enthalpy of hydrolysis of each of the inositol phosphate esters. The values thus far obtained for the diphosphate and higher phosphate esters of inositol show that these esters have low phosphoryl transfer potential and would not therefore be likely to act as phosphagens.

In an earlier section it was stated that studies were made of the enzyme activity of spherosome fractions. They were tested particularly for lipase and for coenzyme A acylase activity. These activities are not associated with spherosomes: the acylase activity is in the soluble fraction and the lipase activity is in a particulate fraction that sediments at the same rate as mitochondria. The lipase found in this fraction differs from the lipase in the resting seed in that the former is inhibited by inorganic phosphate.

Progress has been made in fractionation techniques for seed proteins. This includes improvements in preparative electrophoresis on polyacrylamide gel. Two gels are now being used superimposed one on the other--3% over 5%. Progress has also been made in applying Sephadex fractionation on the seed proteins. Thus, on infrequent occasions it has been possible to isolate a fraction by gel electrophoresis which is pure by criteria of chromatography, gel electrophoresis, and ultracentrifugation. Efforts are now being made to study the conditions under which this is effected and obtain enough of the material for further study.

The proteins of the peanut have been studied by immunoelectrophoresis. It has been found by this technique that purification of arachin or conarachin either by DEAE chromatography or gel electrophoresis changes the mobility of the major fractions. That these are still identical with the original fractions was demonstrated by this technique.

In a previous report it was demonstrated that cottonseed cotyledon shows a difference between the axial portion and distal portion in terms of synthesis of the lipase. Gibberellic acid can promote synthesis of lipase in the distal portion; aflatoxin was shown to inhibit this synthesis. A further study of the effect of concentration of aflatoxin on this property indicates that in low concentrations aflatoxin stimulates the synthesis of proteins in the distal portion; in this respect it has an effect similar to gibberellic acid. Aflatoxin also promotes synthesis of the systems involved in development of chlorophyll pigments; this, too, is a property shown by gibberellic acid. Aflatoxin, however, does not affect elongation of the leaf cells; this property is shown by gibberellic acid. (SU P 1).

B. Flavor

1. Identification of Constituents and Factors Influencing Flavor and Aroma of Processed Products. Investigations of lipid and lipid-soluble constituents of peanuts and their processed products to expand the utilization of

food-grade peanuts have been continued. Attention has recently been directed to the phosphatides and those compounds contributing to the characteristic odor of roasted peanuts. Use of two different solvent systems showed that oils extracted from dry-roasted peanuts have a strong peanut aroma, whereas only a faint aroma is detected in the residual meal; the trace of aroma remaining in the condensate after the solvent is removed is not characteristic of freshly roasted peanuts. Application of a large amount of oil (about 400 mg.) per gram of the silicic acid on chromatographic columns, followed by elution with chloroform-methanol (4:1), revealed that the bulk of the peanut odor is in the predominately cephalin fraction. On careful concentration in vacuo a strong peanutlike aroma occurs in the residue, but a burnt, or over-roasted note is intensified. Over-concentration results in loss of nearly all aroma, which can be detected in the distillate. Since the components responsible for aroma and flavor of roasted peanuts are moderately volatile and are also extractable with the oils, these techniques may have value in relating peanut components to flavor and ultimately to preparing a concentrate suitable for flavoring foods or confections. (S4 1-109(Rev.)).

In completed contract research conducted by Evans Research and Development Corporation, extraction of ground, roasted peanuts with an organic solvent (methanol), and separation of that extract into acidic, phenolic, basic, and neutral fractions, was found to be the most useful method of obtaining flavor and aroma constituents. Each of the different fractions thus obtained had a distinct aroma, usually suggestive of peanuts. Qualitative and quantitative differences noted among the acid fractions of the three varieties of peanuts examined (Virginia, Runner, and Starr) indicate the possibility of genetic control of the flavor of roasted peanuts. The acids identified were acetic, propionic, isobutyric, butyric, isovaleric, valeric, hexanoic, heptanoic, decanoic, lauric, and myristic. Phenylacetic acid was identified, and an unknown acid (possibly a dihydroxynaphthaleneacetic acid) that had no aroma but a bitter taste was isolated. The presence of several phenols was noted in the phenolic fractions, but none were positively identified. Lactones were isolated in small quantities from this fraction. Infrared spectroscopy suggested that these are aliphatic, six-membered lactones. Hexanal, 2, 4-decadienal, and β-sitosterol were isolated from the neutral fractions. Also isolated was a 2, 4-dinitrophenylhydrazone of a compound which appeared to be 2-oxooctanal. Sensory panel evaluations of the peanut extracts showed that a peanutlike aroma was consistently maintained in portions of the neutral fraction. The unique fractionation scheme devised by the contractor may be of value in the utilization of these flavor and aroma components to improve processed peanut products. (S4 1-106(C)).

Other contract work related to the previous project is being conducted by the Agricultural Experiment Station, Oklahoma State University of Agriculture and Applied Science. The relationship of the carbohydrate, amino acid, and protein components of the peanut to the formation of flavor and aroma during roasting is being investigated for shelled and unshelled Argentine variety peanuts cured under four conditions (windrow, 90° F.,

105° F., and 120° F.) and stored under three conditions (ambient, 36° F., and 70° F.). Although the 1964 and 1965 growing seasons were different, each year there were about 66.2% mature and intermediate fruits when the windrow-cured samples were dug. Flavor preference for cured, roasted peanuts before storage was for windrow-cured samples and decreased as the curing temperature increased. Since agreement between replications of analyses on aleurone grains appeared more consistent than those from precursor fractions, these more reproducible analyses may lead to an objective test for evaluating peanut flavor. Consistent differences in ninhydrin (an indication of free amino acids) and pentose sugar values of aleurone grains were noted between peanuts stored shelled and unshelled but these differences could not be correlated with flavor differences. However, it does appear that off-flavor precursors develop during curing rather than during storage, and this lead will be further clarified by planned analyses of selected soluble nitrogen fractions.(S4 1-119(C)).

C. Microbiology and Toxicology

1. Investigation of Occurrence, Determination, and Properties of Fungi and Toxic Fungal Metabolites That May Develop in Peanuts and Their Processed Products. Several lines of research are directed toward solving problems connected with fungi and their toxic metabolites that may occur in peanuts and their processed products. One project, conducted under contract to the Agricultural Experiment Station, Auburn University, concerns the limiting environmental conditions for the elaboration of mycotoxins in peanuts during storage and processing. Both relative humidity and temperature are significant factors influencing aflatoxin elaboration on nonliving peanuts. Limiting relative humidity (RH) for aflatoxin production by A. flavus was found to be 85.5 ± 0.5% RH for 21 days' storage at 30° C. Although equilibrium moisture content of immature kernels was significantly higher than that of sound mature kernels and unshelled nuts, aflatoxin production was essentially equivalent in all cases. Mechanically damaged kernels produced the highest aflatoxin levels. At RH levels of 97-99%, aflatoxins were produced during storage at the relatively low temperatures of 13-20° C. Temperature has pronounced effect on the ratio of aflatoxin B_1 to aflatoxin G_1, low temperature favoring high G_1 and higher temperatures favoring high B_1 production. The limiting maximum temperature at 97-99% RH was 41.5° C. ± 1.5° C.; at 43° C., fungus growth and sporulation were heavy, but no aflatoxins were produced. In chemically defined liquid media, aflatoxins were produced with glucose, sucrose, or fructose as carbon sources. Complex organic nitrogen sources such as yeast extract and peptone produced higher aflatoxin levels than purified amino acids. Little or no aflatoxin was produced when zinc, iron, or magnesium was omitted from the medium, whereas manganese seemed to reduce aflatoxin yields at all concentrations. Plans include the extension of this investigation to freshly dug living peanuts. (S4 1-121(C)).

Much of the work on a related project, in-house research on fungi and toxic fungal metabolites, has recently been devoted to cottonseed. However, the objective densitometer measurements of aflatoxins described in that section

can also be used for purified extracts of peanut products. Research under this project on the processing of peanuts has shown that under standard conditions of alkali refining, all traces of aflatoxins are removed from contaminated peanut oils. Thus, even if contaminated kernels are processed, any toxin carried into the crude oil will be removed during refining. It has also been shown that roasting portions of individual aflatoxin-contaminated peanut cotyledons resulted in an average reduction of 68% in total aflatoxins, with reduction of B_1 greater than B_2. Color is not a good criterion for detecting aflatoxin-contaminated cotyledons either before or after roasting. Work on the effect of roasting on appearance and aflatoxin content of contaminated peanuts will be continued. (S4 1-116).

Peanuts of known history with respect to growing, harvesting, and curing conditions are being studied in contract research conducted by the Agricultural Experiment Station, Texas A&M University; this information will be used in the development of processing methods to produce high-quality peanut products that are free of mycotoxins. Although no data on aflatoxins were reported during this period, additional organoleptic data have been obtained. Peanuts were oil-roasted for 20 minutes at temperatures of 320°, 340°, and 360° F. The 340° product was preferred. Irrigation significantly improved flavor quality, whereas fumigation of the soil did not. Large peanuts, which are retained on a size 17 screen, were of higher flavor quality than smaller kernels. Oil-roasted peanuts processed from bag-dried fruits were preferred to those prepared from field-dried. Oven-dried kernels yielded the lowest flavor scores, but differences were small, as was the case with dry-roasted peanuts. These findings may assist in selecting raw peanuts for processing into edible products of highest quality: larger size, grown under irrigation and cured in bags at ambient temperatures after threshing immediately following harvest. (S4 1-120(C)).

Exploratory work on the inactivation or removal of aflatoxins from contaminated peanuts and their products has resulted in advances that may ultimately permit their utilization in foods and feeds. Treatment of peanut meal with ammonia or with aqueous acetone may offer a practical means of reducing its aflatoxin content. Peanut meal containing approximately 3100 ppb of B_1 was adjusted to 15% moisture and treated with anhydrous ammonia under 40 psig pressure for about 16 min. at a maximum temperature of 178° F. The aflatoxin content was reduced to 17 ppb. Aqueous acetone (5 to 10% water) extraction of peanut meal containing 60 ppb B_1 reduced the content 80-85%. In contrast, treatment of peanut meals with amines was less effective than with the previously mentioned agents. Peanut meal containing 60 ppb B_1 was adjusted to 15% moisture, treated with methylamine, and stored in sealed containers at elevated temperatures. After 24 hrs. at 38° C., there was only slight reduction in aflatoxin content; after 3 hrs. at 93° C. aflatoxin was reduced 75%. Plans include the investigation of various chemical treatments on both a laboratory and pilot-plant scale to develop practical methods for inactivating aflatoxin. (S4 1-133).

D. Technology -- Process and Product Development

1. Peanut Flours and Derived Products for Human Consumption in Developing Countries. Peanut meals of high quality, judged by chemical analyses, have been prepared under a project supported by the Agency for International Development (AID). A high-quality, low-fat peanut flour was prepared for evaluation by Human Nutrition Research Division (HN); results were good to excellent in such diverse preparations as beverages for babies, cookies, breads, chapatis, curry, garbanzo stew, pancakes, and noodles. Peanut meals and flours have also been shipped to Canada, England, and Harvard University for various investigations of their nutritional values. Plans include development of the simplest and most practical processes for relatively small plants in developing countries to use in making highest quality peanut flour; continued improvement of the flour; and evaluation of medium-fat peanut flours for food use. (SU-0-0-3(AID)).

2. New Processed Products, Including Partially Defatted Peanut Products. Based on information developed at SU, commercialization of the partially defatted peanut product has been realized. One company is now producing and market testing, on a large scale, low-fat Spanish peanuts in several areas in the South. The product is sold in 10¢ flexible opaque bags. Another company is now offering pressed Virginia peanuts in sufficient quantities for development purposes or for use in preparing low-fat peanuts for the retail market. Many other companies are developing and evaluating low-fat peanut products, and several are planning market tests soon. New and delicious spice-coated low-fat peanut products have been developed. New information on the low-fat peanut product development is continually being made available to industry. Cooperative investigations with processing equipment manufacturers to upscale the process for commercial application continued. Pilot-plant runs conducted with two manufacturers show that: (1) commercial fluid-bed dryers are suitable for the production of dry-roasted low-fat peanuts, and (2) further development work is needed before infrared drying equipment can be used for this purpose. Shelf-life studies are being conducted on products produced by these processes. Large-scale investigations with commercial cage presses (with 20" diameters and 24"-deep material-holding chambers) show less oil removal than that obtained for the same pressures in laboratory and pilot-plant size presses with cake thickness of 3". Bench and pilot-plant scale work to investigate and improve specific operations of the process also continued. Tests with peanuts having low-moisture content (1-4%), expansion with a puffing gun, and pressing but not expanding in hot water did not give satisfactory results. However, preliminary work showed that L-lysine monohydrochloride can be added internally to low-fat peanuts during expansion; practical methods for this addition and evaluation of the resulting products will be studied, as well as other improvements in properties and processing. (S4 1-126).

Studies continued on preparation of defatted peanut flours, meals, and grits, as specified in the research contract awarded to the Agricultural Experiment Station at Auburn University. Screw pressing operation was

improved by use of radiant heat, but not by reducing screw speed. Results of lye blanching tests appeared very promising. Exposure to lye for only 20 seconds removed 100% of the skins with little separation of kernel halves. Blanched peanuts had a bright glossy appearance. The problem of grinding to a flour fineness was solved by use of a recently acquired special mill. Methods were tested for solvent extraction of partially defatted peanuts and for detection of hexane residue. Products were prepared for recipe tests. Shelf-life studies were begun on several products in glass and in saran. Work was initiated on use of peanut products in ice cream and confections. A combination of peanuts and applesauce, drum dried and used in a mix, has produced a good product. Raw peanut products were found to be definitely unacceptable because of their undesirable flavor and aroma, which remained despite additions of various flavorings. Meals and grits in the lower oil levels were unacceptable because they retain a hard mealy or gritty texture. Meals and grits at all oil levels were unacceptable, in products such as soups, gruels, dips, ice cream, and puddings because of their gritty texture. Meals at three higher oil levels can be used in some baked products, such as cookies and the "heavier" cakes, e.g., nut breads. Plans include the continued preparation of peanut products at different oil levels and the evaluation of their properties. (S4 1-118(C)).

3. Methods Developed for Inactivating or Removing Aflatoxin from Contaminated Peanut Kernels. The development of processing conditions to produce optimum-quality, mycotoxin-free roasted peanuts is the objective of a contract awarded to the Agricultural Experiment Station, Oklahoma State University. Three preliminary areas are presently being investigated. In the first area, field experiments were conducted with the Argentine variety of Spanish-type peanuts. A. flavus spores were inoculated onto freshly dug peanuts; variables studied included presence or absence of plastic coverings, treatment with chemical inhibitors, and two field incubation periods. Samples of freshly dug peanuts were stored at 20° F. All field readings--regardless of inoculation, plastic coverings, or chemical treatment--were negative for observed pod surface fungus development at both incubation times. The cold storage samples are being used in laboratory incubation studies at 30° C. in saturated humidity atmosphere. In the second area, work was initiated on the design of a laboratory vacuum drying system. In the third area, means of analyses for aflatoxin have been set up and will be used on selected samples from the field trials. (S4 1-132(C)).

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AREA 6 - CITRUS AND SUBTROPICAL FRUIT UTILIZATION - FOOD

Problem. The citrus and subtropical fruit production of the Southern Region is an expanding industry with the need for the development of better, as well as new-type consumer products, and for the improvement of present or invention of new processing procedures and machinery. These advances are required to regularly utilize the currently large production, particularly of oranges and grapefruit, and the anticipated higher production of these fruits, to the economic advantage of the growers and consumers. Basic research is needed to lay the groundwork for these advances. This research is needed, for example, on the composition and physical nature of essential oils, flavonoids, including bitter constituents, constituents responsible for oxidized off-flavors, carotenoids, and the like, which determine many of the sensory characteristics, and which affect product quality and stability. Other problems whose solutions are dependent upon the availability of more detailed compositional and physical data are: cloud stability, gelation, discoloration, fermentation, and the like. Increased production of citrus has stimulated the development of new products, but many of these are urgently in need of improvement which will depend in part upon advances in basic research. New products are needed to attract new markets and also to reduce packaging and shipping costs. Research is needed to improve frozen citrus concentrates as processing procedures change; to develop better high density concentrate products, citrus powders, chilled juice and section products, pulp-fortified products; comminuted whole fruit products; and to develop new or improved canned products which have a natural fruit flavor. Research is especially needed on grapefruit to develop practical methods for reducing the bitterness and harshness of juice products and to increase the use of grapefruit juice base in mixed fruit juice blends, drinks, concentrates, and the like. Along with progress on product development there is a serious need to improve the actual processing procedures, processing equipment, and packaging operations and materials, to obtain and maintain the most desirable fruit characteristics particularly for citrus powders. In addition to the work on citrus, research is currently needed to develop new processed products from avocados.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, and a chemical engineer engaged in both basic and applied utilization research studies on citrus and subtropical fruits of the Southern Region to develop new or extended uses for these commodities.

Research to develop basic information on the chemistry of flavor of citrus and subtropical fruits and their products and byproducts is conducted at the U. S. Fruit and Vegetable Products Laboratory at Weslaco, Texas, and Winter Haven, Florida. This information provides the necessary basis for efficient research in developing new and improved food products and processing technology. At the Weslaco Laboratory, the program includes

investigations of the influence of seasonal changes of carotenoid and flavonoid constituents which directly or indirectly affect flavor and color of processed products from Texas colored grapefruit, as a basis for improvement of processing characteristics of and products from these grapefruit. The Texas Agricultural Experiment Station (substation 15, Weslaco), Citrus Rootstock Investigations Laboratory (CR, ARS, Weslaco), the Texas College of Arts and Industries, and Rio Farms, Inc. (Edcouch) are providing grapefruit of known history and conducting, or cooperating in conducting, on-the-tree tests. At the Winter Haven Laboratory, the program includes: research to identify recently isolated flavones and other neutral orange peel constituents and to evaluate their relation to bitterness and harshness in orange products; investigations of the composition of essential oils in citrus products, particularly orange, to provide a basis for improvement in quality and uniformity of citrus products; a study of off-flavor development in processed citrus juice in relation to the lipid composition of the suspended matter; and research to explore means for minimizing or blocking the formation of bitter components in grapefruit, a key step in developing processed grapefruit products of greater attractiveness to the consumer. Close consultation is maintained with the Florida Citrus Commission (Lakeland); the Florida Agricultural Experiment Station (Citrus Experiment Station, Lake Alfred); Citrus Research Investigations (CR, ARS, Orlando); Florida Citrus Mutual (Lakeland); and the citrus processing industry.

Research to develop new and improved process and product technology is carried out at the U. S. Fruit and Vegetable Products Laboratories at Weslaco, Texas, and Winter Haven, Florida. At Weslaco, research is in progress to develop processed products from grapefruit and from avocados. The citrus and subtropical fruit research is being carried out in part in cooperation with several state and private organizations. The cooperators provide fruit or raw materials, such as pulp and juice, of known history. Processing plant facilities are available from the Texsun Citrus Corporation (Weslaco) and Rio-Vac, Inc. (Harlingen). Formal agreements exist with the Texas Agricultural Station (College Station and Weslaco), with Texsun Citrus Corporation (Weslaco), and with Rio Farms, Inc. (Edcouch). Informal cooperation is maintained with Texas Citrus Mutual, Inc. (Weslaco), Texas Canners Association (Weslaco), and such other organizations as necessary for the procurement and processing of fruit. At Winter Haven, conditions for the "foam-mat" type of air-drying as they relate to the storage stability and quality of the resulting citrus products are being studied. This research is conducted in cooperation with the Western Utilization Research and Development Division (ARS) and the Florida Citrus Commission under a formal memorandum of understanding. Contract research on process and product development, supervised by the Weslaco Laboratory, is being carried out at the Citrus Experiment Station, University of Florida, Lake Alfred, Florida. It pertains to the development of a practical and efficient pilot plant scale process for the production of enzymatically debittered grapefruit juice and products with improved flavor, product stability, and storage characteristics.

The Federal in-house scientific effort at the Southern Division devoted to work in this area totals 11.9 scientific man-years. Of this total, 7.3 is devoted to research on flavor and 4.6 to technology-process and product development. The contract research involves an additional 0.3 scientific man-years on technology-process and product development.

The only line of work to be terminated during the year was the investigation of the effect of maturity of grapefruit on total flavonoids, naringin, and poncirin, and of the chemistry and nature of naringin and naringin-derived compounds to provide a scientific basis for the control of bitterness in processed grapefruit products (under Flavor).

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 19.0 scientific man-years is devoted to citrus and subtropical fruit utilization research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Flavor

1. Chemical and Physical Properties of Flavoring Constituents of Florida Citrus and Subtropical Fruit Products. In the investigation of essential oils in citrus products, the alcohol analysis for Florida cold-pressed oils of grapefruit, lemon, lime, and tangerine has been completed. In addition to the 17 alcohols previously identified, three other alcohols were found in grapefruit. However, the presence of one of these compounds, o-phenyl phenol, may be explained by its use as a fungicide by the citrus industry. Since the alcohols in orange, grapefruit, and tangerine are very similar, the distinctive differences between the oils would not be expected in this fraction. The alcohols in lime and lemon are almost identical.

A procedure has also been developed for the separation of oxygenates other than alcohols. Although this procedure has not yet been perfected, it has been applied to orange cold-pressed oil with a good deal of success. It includes low temperature, reduced pressure stripping of limonene; column chromatography for the separation of the oxygenated group; and gas chromatography for isolation of individual components. In another facet of the research, gas chromatographically pure nootkatone was obtained from (1) cold-pressed grapefruit oil, and (2) the synthesis of nootkatone from valencene. The pure nootkatone was used in taste tests to determine its effect on the flavor in grapefruit juice. In addition to the above progress, mass spectra of all compounds submitted by the laboratory staff have been obtained. (S3 2-48).

2. Investigation of the Bitter Principle and Flavonoids in Citrus Products. In the continuing work on bitterness in oranges, an analytical experiment showed that the five flavones referred to in previous reports occurred in both the serum and the suspended matter of the concentrate investigated, some being more concentrated in one phase and some in the other. Evidently,

all of the flavor due to the neutral fraction can be accounted for by the flavones in the late season fruit but not in the early season fruit — re-evaluation of taste thresholds of the fractions gave the following results: December 1963, 17; January 1964, 23; March 1964, 29; early April 1964, 33; late April 1964, 31; and June 1964, 30. The five flavones gave threshold values as follows in ppm: tangeretin, 33; heptamethoxyflavone, 28; nobiletin, 46; sinensetin, 30; and tetra-O-methylscutellarein, 15. All the above evaluations were carried out in a synthetic medium employed because of its reproducibility and the economy of flavones that could be detected at a lower level than in orange juice. These values should be useful in estimating the relative amounts required for evaluations of the compounds in orange juice and will also serve as a guide in later work. (S3 2-47).

Considerable progress has been made by the contractor, University of Oklahoma Research Institute, in determining the effect of grapefruit maturity on total flavonoids and on naringin and poncirin. In the 1964-65 season, the monthly variation in concentration of six flavonone glycosides in the juice sacs of Texas ruby red grapefruit was determined. These glycosides are naringin, naringenin rutinoside, neohesperidin, hesperidin, poncirin, and isosakuranetin rutinoside. The total flavonoid concentration decreased with maturity, but the ratio of bitter to nonbitter flavonones remained relatively constant. This finding failed to support a previously reported hypothesis that the decrease in bitterness of grapefruit may be the result of a transglycosylation that changes the bitter flavonones (7-β-neohesperidosides) to tasteless (7-β-rutinosides). Davis test values made on the same samples of fruit show a close correlation with the quantitative data for the flavonone glycosides, particularly the principal ones, naringin and naringenin rutinoside. Although the data have not yet been completely analyzed, they may indicate that bitter compounds other than naringin are involved in the decrease in bitterness of grapefruit with maturation, or that a nonbitter compound which appears in larger quantities in maturing fruit "masks" the bitterness of naringin. (S3 2-39(C) (Rev.)).

In research directed toward minimizing bitterness in grapefruit products, a new and more rapid method for the determination of naringin in grapefruit juice was developed. The results are available in four hours instead of the two days previously required, and the procedure is simple enough to be used by plant personnel. It is a modification of earlier procedures used for the separation of naringin (bitter) and naringenin-7β-rutinoside (tasteless), followed by their individual determination colorimetrically. The new method will aid in determining the effect of various possible inhibitors on the synthesis of naringin and on the seasonal variation of both constituents. It can also be used by the citrus industry to give an accurate and objective determination of naringin concentrations to be compared against subjective taste evaluations. Other work under this project showed that phenylalanine is a precursor to naringin in grapefruit. This was demonstrated by implantation of the radioprecursor into the phloem area of the stems, a technique that presently appears to be the

best means of getting labeled solids into the grapefruit plant. (S3 2-49).

Considerable progress has been made in a relatively new project designed to determine the influence of seasonal variations in flavor and color of Texas grapefruit on the quality of processed products. The study of the effects of environmental temperature on lycopene content of grapefruit has been completed. Grafted grapefruit on trees maintained in 95/85° F. day/night chamber remained green and increased in lycopene content throughout the 20-week period. Fruit in the 70/60° F. chamber lost its green color, yellowed, and declined sharply in lycopene content. Control fruit went through a normal increase and decline cycle. Establishing the fact that temperature is a major environmental factor in influencing the formation and disappearance of lycopene provides fundamental information by which growers and processors may be guided in the utilization of fruit at a time of optimum color.

Completion of tracer studies on grapefruit treated with $C^{14}O_2$ radioactive carbon dioxide at different times during the 1964-65 season has bracketed the period of maximum flavanone production as occurring during the first six weeks of fruit growth. More intensive tests are now in progress to evaluate quantitatively the period of flavanone formation. Improved methods of preparative and quantitative flavanone column chromatography that facilitate exact compositional studies have been developed. A number of minor polyphenol fractions that may influence processing and quality characteristics of grapefruit products have been found. Evaluation of a number of resins for column chromatography has shown polyvinyl polypyrrolidone to be superior for flavanone separation. Carbon was unsuitable because of incomplete recovery of absorbed materials. The completion of flavanone analysis of fruit from trees treated with a paraffinic oil spray has established that this cultural practice has no influence on flavanone composition of grapefruit. A method for the quantitative recovery of the limonoid compounds in grapefruit has been developed, employing continuous counter-current chloroform extraction of the depulped juice. The limonoids in the pulp are extracted separately with acetone. Taste tests on authentic limonin indicates that it contributes little to the total bitterness of Texas grapefruit products. Determination of various factors affecting flavonoid contents should lead to an improvement in characteristics of products made from colored grapefruit. (S3 2-51).

B. Technology -- Process and Product Development

1. Application of Foam-Mat Drying to Citrus. In research conducted in cooperation with WU and the Florida Citrus Commission, drying conditions of foam-mat powders are being studied with respect to storage stability and quality. Orange crystals made from concentrate which contained oil have a slightly better storage life than those made from concentrate without oil. Both develop detectable change in flavor in 4 weeks at 85° F. This coupled with analytical tests indicates that the oil acts primarily as a covering-flavor agent and does not retard the adverse

reactions occurring during storage. Several other furan and furfural type derivatives have been isolated and identified from "off-flavor" orange powder, providing more evidence of nonenzymic browning type reactions. Combinations of orange and grapefruit have resulted in crystal samples which have storage life improved over that of orange alone. Actual reasons for this increased stability are still being investigated. It has been found that algin derivatives are very useful for addition to low-viscosity orange concentrates to make them more suitable for foaming. This would indicate that viscosity is strongly related to the foaming characteristics of a concentrate. Expressions of interest from several commercial developers were evoked by promotional distribution of packets of grapefruit powder in an industry magazine. (S3 2-43).

2. Process for Enzymatically Debittering Grapefruit Products. After a six-month suspension of activity on contract research conducted on a pilot-plant process for enzymatically debittering grapefruit juice, work has again begun as bitter grapefruit have come into season. Experiments were conducted to determine the effect of high sugar concentration and sodium benzoate on the activity of naringinase. To commercial grapefruit sections, a 10% sodium benzoate and 40° Brix syrup was added. To this mixture, 0.1% naringinase was added and the product was held at 32° F. The presence of preservative on sugar syrup did not significantly interfere with the activity of the enzyme as it reduced naringin. Over 60% of the bitterness was removed after 5 days' storage and 79% reduction in naringin level occurred in about 30 days. Action on naringin was slower when lower levels of naringinase were added, but with only .005% naringinase, 10 days' storage reduced naringin by about 30% and 34 days' storage by slightly over 50%. Storage studies on these samples are continuing. Work has also continued on enzymatic debittering of pulp washed solids. Although naringin declined more slowly at cold storage temperatures than at high temperature, it is evident that addition of small amounts of naringinase (.005 - .010%) to products that are customarily stored for a number of days will cause a significant decrease in bitterness. (S3 2-46(C)).

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AREA 7 - VEGETABLE UTILIZATION - FOOD

Problem. Although extensive progress has been made in recent years in developing stable, attractive, and convenient to use vegetable products, new and improved processed products must be developed and means of stabilizing perishable vegetables provided to minimize the adverse effects of seasonable surpluses and unfavorable markets, and to provide an adequate supply of good food for a growing population. Product quality needs to be improved and processing cost reduced through the adaptation and application of the latest technological developments and nutritional findings. For example, a major problem of the cucumber industry, since most of the crop is brine-cured, is to improve the curing process so that no loss occurs in the value of the cucumber during the brine-curing and storage process and the cost of processing is reduced. New pure culture fermented products are needed to more fully utilize cucumbers and many other vegetables in attractive consumer items. As another example, a precooked, dehydrated, sweetpotato product has been developed which usually has good shelf life when sealed under an inert gas. It reconstitutes to a product having the characteristics of freshly cooked and pureed sweetpotatoes. Applied research on a pilot-plant scale is needed to obtain additional engineering and processing data applicable to commercial production of flakes from sweetpotatoes of different variety and environmental history. Basic research is needed to further improve quality and storageability of the product, and to provide the scientific basis for the development of a process for making excellent flakes from uncured, freshly dug sweetpotatoes. There is a continuing need in the use of vegetables for processing to investigate the characteristics of the raw material as these characteristics are affected by climate, soil, cultural practices, breeding, and the like. Celery, already an important flavoring ingredient, could become much more important if the factors and constituents responsible for the intensity, variableness, and stability of its flavor could be controlled in processing, and processed products of improved flavor and convenience could be developed. Many vegetables grown in the Southern Region differ in their chemical and physical characteristics from the same crops grown in the more temperate regions. Tomatoes are a good example in that they are frequently poorer in color, flavor and texture. Several vegetable crops, including sweetpotatoes, hot peppers, okra, and Southern peas, are grown almost exclusively in the Southern Region. More utilization research is needed to complement the Federal and State production research programs and to provide cooperation in the form of composition and processing studies. This kind of cooperation is needed to prevent the release of breeding selections which are entirely unsuited for processing.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, microbiologists, food technologists, and chemical

engineers engaged in both basic and applied utilization research studies on vegetables of the Southern Region to develop new or extended uses for these commodities.

Research to develop basic information on chemical composition and physical properties of vegetables, their products and byproducts, is conducted as a basis for efficient research in developing new and improved food products and processing technology. Research conducted at and in cooperation with the North Carolina Agricultural Experiment Station, Raleigh, North Carolina, is concerned with basic investigations of the chemistry and biochemistry of the carotenoid pigments in vegetables in relation to variety, maturity, and environmental factors, to facilitate the development of improved and more attractive processed products. Additional research on chemical composition and physical properties is being carried out under a grant at the Research Triangle Institute, Durham, North Carolina, on elucidation of the molecular structure and chemical characteristics of the pectinase inhibitor that occurs in sericea forage and other plant sources and has proven effective in preventing softening of cucumbers in brine curing.

Investigations of the effects of cucumber substrate, bacterial species, and other environmental factors on the flavor and aroma components of natural and pure culture fermented cucumber pickle products are carried out at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina, to provide the basis for producing pickle products of greater consumer acceptability. The North Carolina and Michigan Agricultural Experiment Stations, and the Pickle Packers International, Inc., cooperate in this research.

In the development of technology for new and improved processes and products, both basic and applied research is being carried out at New Orleans, Louisiana, to improve the stability of the flavor of precooked, dehydrated sweetpotato flakes packaged in air, and to improve the processability of uncured sweetpotatoes and their flake characteristics. These are two major problems still facing the new sweetpotato flake industry. Current research approaches involve the use of sweetpotatoes in combination with other foods to produce new products, the incorporation of nutrients, and investigation of constituent changes produced by the enzyme α-amylase. Pilot-plant investigations will also be conducted as a phase of this research. Cooperation is maintained with the Marketing Economics Division, ERS, for the market evaluation of improved flake products, and with the Louisiana Agricultural Experiment Station, the Louisiana Sweetpotato Association, the Louisiana Sweetpotato Commission, and various industrial concerns. Other research on process and product development is in progress at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina. Current emphasis is on investigations of methods for the controlled fermentation of cucumbers and other vegetables by application of pure culture techniques to fermentation practices in order to reduce processing costs and improve product characteristics. Limited cooperative work is conducted to evaluate new cucumber varieties (or selections) for processing into brine-cured and fresh-pack products. Cooperation is maintained with the North Carolina

Agricultural Experiment Station. The Michigan State University (Department of Microbiology) is also cooperating by providing technical assistance in the controlled fermentation studies. The Pickle Packers International, Inc., contributes support to the research and supplies raw material. The U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas, is conducting research directed toward developing new and improved processed products from southern grown vegetables other than sweetpotatoes and celery. The Texas Agricultural Experiment Station, the Crops Research Division, ARS, and industry associations provide raw materials of known history for this research. In progress at the U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, is research on the development of processed celery products of improved flavor and convenience.

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 9.4 scientific man-years. Of this total, 2.2 is devoted to chemical composition and physical properties, 1.3 to flavor, and 5.9 to technology-process and product development. The domestic grant research involves an additional 0.6 man-years, on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 48.0 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Investigations of the Chemistry and Biochemistry of the Carotenoid Pigments in Vegetables. In cooperative research with the North Carolina Agricultural Experiment Station on the chemistry and biochemistry of the carotenoid pigments in vegetables and fruits, the study of the carotene which stimulates photo-reduction of DPN has continued. This pigment has been reproducibly isolated from fresh spinach. Its spectrum does not show the fine structure characteristics of most carotenoids. Examination of a fraction of the pigment, believed to be the purest yet obtained, by infrared spectroscopy indicated that the compound is probably a poly isoprene with a carbonyl. The presence of a carbonyl is surprising since it was previously found that reduction with lithium aluminum hydride did not completely destroy activity. Raw and cooked carrots, sweetpotatoes, and tomatoes, all of which are high in carotenoids, have been examined with a spectrophotometer; in all cases, the carotenoid absorption spectra of the cooked slices shift toward lower wavelengths. This shift indicates a change in the physical state of the pigment. Impure chromoplast preparations undergo the same changes. Microscopic examination has indicated a loss of organization when a marked spectral shift occurs. This applies to chromoplast in the slice, isolated chromoplast, and suspended crystals. When the organization is thus disrupted by cooking, the carotene can be dissolved in other lipids. This change causes visual yellowing of the

product without the loss of pigment. It is probable that changes in the stability of the carotenes occur as their physical state changes. If this can be shown, spectral and microscopic methods may be useful for defining optimum processing conditions for obtaining more stable products, with respect to both flavor and color. Also, verification and exploitation of these preliminary observations should provide information which will guide processors in improving the color in processed products. (S3 5-28).

2. Identification and Characterization of Inhibitor of Enzyme that Softens Cucumbers. Under a grant with the Research Triangle Institute, the molecular structure and chemical characteristics of the pectinase inhibitor in sericea are being investigated. The first significant purification of the pectinase inhibitor beyond the caffeine method has been achieved by countercurrent distribution (CCD) systems. Several solvent systems were tested for their ability to fractionate the crude inhibitor material, but it soon became apparent that both phases of the CCD system would need to be highly polar. The results of preliminary tests with a neutral solvent system (2-propanol-water-sodium chloride) were promising enough to warrant a 200-tube CCD experiment, which was carried out on a 1.6 g. inhibitor sample. Two major fractions were separated; based on pectinase enzyme inhibition, the first fraction gave a very high purity with a freeze-dried yield of 0.265 g. The fourfold purified fraction was found to be less stable than the initial sample, a difference which suggested that a natural protecting agent (e.g. antioxidant) had been removed in purification. Since the inhibitor is a pro-anthocyanidin, the rates of pigment formation were given further study. Under controlled reactions, it was found that atmospheric oxygen and nitrogen had no effect, that benzoyl peroxide (radical initiator) increased color formation with destruction of the pigment, and that ferric chloride with HCl increased both the rate and final yield of the pigment. This basic information on the inhibitor reactions to form pigment should assist in a closer identity of the molecular structure with possible pathways to synthesis of the inhibitor substance. This is an important step in the commercial use of such material in the control of enzymatic softening of brined cucumbers. (S3 5-24(Gr)).

B. Flavor

1. Factors Affecting the Flavor and Aroma of Cucumbers and Fermented Cucumber Products. Basic studies of the flavor of cucumbers and fermented cucumber products are continuing in cooperation with the Pickle Packers International, Inc., and the North Carolina and Michigan Agricultural Experiment Stations. Direct headspace vapor analysis by gas-liquid chromatography (GLC) as a means of determining volatile components of fermented vegetables is being investigated. Data collected on 24 samples of fermented vegetables indicate that the "headspace" technique is reproducible, provided that factors influencing vapor pressure are standardized and that GLC profile data are related to a suitable internal standard (2-butanol in present case). Evaluation of the data indicated that the fermenting organism (of the three - Lactobacillus plantarum, Pediococcus cerevisiae,

Leuconostoc mesenteroides) can be predicted from GLC data on the basis of quantitative but not qualitative differences. The same volatile components seem to be present, even in acidified, nonfermented controls, but vary in concentration. Several methods are being used to identify these compounds. Findings thus far include: (1) acetaldehyde concentration is reduced by all three fermentations as compared to the acidified control, but most by L. plantarum; (2) a higher concentration of ethyl formate (tentative identification) occurred in the L. plantarum fermentation; (3) ethanol is much higher in the L. mesenteroides fermentation, as expected for a heterolactic organism; (4) several of the same compounds are present in similar amounts in both fermented and acidified products, and probably are not a function of the fermentation. Headspace vapor analysis is faster and gives better quantitative relationship of components than does the distillation procedure customarily used for qualitative identification of volatile components of fermented vegetables; however, a disadvantage is that only those compounds which are present in relatively higher concentration and have a sufficiently high vapor pressure can be measured. (S3 5-29).

C. Technology -- Process and Product Development

1. Investigations to Improve Quality and Lower Costs of Processed Cucumbers and Other Vegetable Products. Since there is an increasing need to ship cucumbers long distances before they are used for fresh-pack pickles and brine-stock, their quality must be preserved for several days. This problem is being investigated in a study of the pure culture fermentation of vegetable products, research that is also conducted in cooperation with the Pickle Packers International, Inc., and the Michigan and North Carolina Agricultural Experiment Stations. The increase in pectinolytic and cellulolytic enzyme activity on cucumbers during refrigerated storage was observed in earlier experiments. The hydrolytic enzymes were attributed mainly to increase in fungal growth on the cucumber. Bacteria are known to grow at refrigeration temperatures and produce pectinase. Two experiments using Model variety cucumbers were carried out under laboratory conditions during the 1965 growing season. These limited studies indicate that microbial deterioration is rapid (1 to 3 days) at 60° F. and 85° F. whereas the 40° and 50° F. lots showed little or no mold. Moreover it took 4 to 10 days for serious deterioration to occur at the lower temperatures. The degree of microbial activity is not always readily visible. Tests of pectinolytic activity and of cellulolytic activity indicated that of the growth media tested, the one showing cellulolytic activity appears to offer the most promise for indicating the degree of deterioration of the vegetable at a given holding temperature. This information has direct practical value for shippers, truckers, and processors. (S3 5-27).

Cooperative research with the Texas Agricultural Experiment Station to improve food products processed from southern-grown vegetables, including carrots and tomatoes, has continued. Completed storage studies have demonstrated that precooked dehydrated carrot flakes packaged in nitrogen

can be stored at 68° F. for as long as 24 months with little loss in quality. However, taste evaluations of air packs were discontinued at the end of three months because of poor flavor of the flakes. Unfortunately, the present yield by the double drum drying process appears to be too low to encourage commercialization of the process for making the carrot flakes.

Two practical pieces of information about tomatoes have resulted from recent research on improving processed products from Southern-grown vegetables. The effect of heat on the viscosity of tomato juice was analyzed for Chico, La Bonita, M-66, and Homestead varieties. When the juices were held at 190° F. for 2 hours, the viscosity of all except the last rapidly dropped, then remained constant; the viscosity of Homestead, very low initially, remained at the same level throughout the test. The concept that the viscosity of tomato juice will decrease rapidly when it is held for prolonged periods at a pasteurizing (canning) temperature is not new; however, these convincing specific data should encourage Texas canners to speed up their processing lines to achieve a quality pack of juice or puree. The second practical preliminary result is that tomatoes peeled in a $CaCl_2$ solution are as firm as tomatoes canned with a $CaCl_2$ tablet added to the can, but the $CaCl_2$ peeled tomatoes contain only about 1/3 as much calcium. Moreover, firming at the time of peeling may reduce loss in the processing line. To date, however, a satisfactory method has not been developed for recovery and reuse of the calcium chloride from contaminated blanch water. This project has now been discontinued, but leads on this phase of the research will be developed in a new project, also to be conducted in cooperation with the Texas Agricultural Experiment Station. (S3 5-22).

2. New and Improved Dehydrated Sweetpotato Products and Processes. The research to develop stable sweetpotato flakes from different varieties of sweetpotatoes has continued. The first phase concerns further study of the high temperature-tolerant α-amylase found in sweetpotatoes. In addition to achieving confirmation of previous results, recent research has shown that the enzyme increases during curing and storage. This is significant because it poses a continually changing seasonal problem in its control, as it converts starch rapidly when raw ground sweetpotatoes are cooked preparatory to making flakes. The addition of calcium salt enhances its activity. This enzyme can be activated in early season as well as late season roots by steam injection heating or indirect heating of sweetpotato puree, or by heating pieces (sliced or diced) of sweetpotato in water. Flakes produced by the latter method have an improved texture and more natural sweetpotato flavor when they are reconstituted. These observations now make it possible to prepare an acceptable flake product from the Goldrush variety under conditions of curing and storage studied to date.

The second part of this research consists of exploratory investigations of the preparation of new food products from sweetpotatoes. Pleasant tasting new drum-dried flake products have been prepared from sweetpotato puree

mixed with apple and peach purees. Also, pleasant tasting protein-enriched dehydrated products have been prepared from sweetpotato puree mixed with various types of peanut flours. Mixing a sweetpotato flour (prepared from flakes) with a low viscosity peanut butter has resulted in new products with consistencies varying from peanut butterlike to cheeselike. A study has also been initiated to determine factors affecting the bulk density of sweetpotato flakes. (S3 5-25).

3. Development of Processed Celery Products of Improved Flavor and Convenience. Significant progress has been made in research directed toward improving celery products. One problem had been that lack of uniformity in the moisture content of celery partially dried for explosion puffing prevents maximum rehydration of the dehydrated product. Pressing blanched, frozen, and thawed celery gave a much more uniform product for puffing than did pre-drying, and also considerably improved rehydration. When used in conjunction with other pre-drying treatments, pressing permitted the production of dehydrated celery that absorbed over 35 times its weight of cold water. The rehydrated products from such multiple treatments resembled freshly cooked celery in appearance and texture but they lacked flavor. A systematic study of several pre-drying treatments — including blanching, freezing and thawing, pressing, and leaching — was begun to clarify the rehydration process. Preliminary conclusions indicate that different pre-drying treatments may require different methods of rehydration to give best results.

Since it will be necessary to fortify the flavor of these products before they are marketable, it is encouraging that a practical method for producing essential celery oil for flavor fortification of dehydrated celery has been devised. It is based on steam distillation of raw celery material and rectification of the condensate. Adequate quantities of the oil can be obtained from leaves and trimmings normally wasted in harvesting celery. Preliminary studies of the composition of the essential oil obtained by rectification indicate that many more compounds, including additional phthalides, are present than previously found in solvent extracted oils. Several of the compounds isolated thus far that possess the characteristic celery odor have not yet been identified. Thus, the chemistry of celery flavor appears to be more complex than was first thought. It is desirable that the compounds in the oil be isolated and examined to determine their respective contributions to celery flavor. Work was also initiated on the development of a micro-method for determining the amount of oil present in fresh, raw celery, based on major flavor contributing phthalides. (S3 5-23(Rev.)).

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AREA 8 - NAVAL STORES UTILIZATION - INDUSTRIAL PRODUCTS

Problem. More uses for pine gum, rosin and turpentine need to be developed through research to provide industrial markets for current and anticipated production of gum naval stores. Recent decreases in uses of gum rosin, especially in paper size, has resulted in the accumulation of a considerable surplus of this commodity. Other types of rosin as well as synthetic chemicals backed by strong industrial research programs have made serious inroads on the traditional markets for gum rosin. Gum turpentine is also faced with similar competition. If the turpentine farmers of the southeast are to continue to find profitable markets for their pine gum, existing knowledge of the properties of this commodity and its derived products must be used to develop new uses and strengthen old ones. New fundamental information about the chemistry of the terpenes and resin acids is also needed to fully exploit their unique characteristics. New or expanded uses for naval stores products are especially needed in polymers, plastics, elastomers, resins, plasticizers, surface coatings, textile finishes, odorants, insecticides, herbicides, and other large-volume industrial chemicals. There is also a serious need to improve existing processes and develop new processing technology for the industry.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program at Olustee, Florida, involving organic chemists and a chemical engineer engaged in both basic and applied research to discover and develop new and improved uses for pine gum and its products. In research to develop new and improved industrial products from pine gum, rosin, turpentine, or their components, conversion of the resin acids derived from gum rosin and pine gum to new polyfunctional products by reaction with suitable chemicals is under investigation to develop intermediates for production of resins, plastics, and related products. Another research approach involves the condensation of the unsaturated (olefinic) materials present in pine gum with certain reactive chemicals to produce industrially useful chemicals. Research is being conducted to develop improved polyester resins from resin acids of pine gum and rosin; to study the preparation and reactions of epoxides and ozonization products of resin acids and their derivatives to produce materials having potential for use in plastics and other industrial products; to develop practical methods for converting levopimaric acid, resin acid mixtures; and/or pine gum to polyfunctional compounds useful in plastics, resins, and other industrial products by formaldehyde addition and subsequent reactions; to convert terpene acids, terpene acid derivatives, and rosin derivatives into polymerizable monomers suitable for making new polymers, plastics, and resins; and to produce reactive chemical intermediates from turpentine and terpenes derived from it. Informal cooperation is maintained with other agencies and industrial firms in connection with the naval stores research program. The U. S. Forest Service cooperates by supplying selected samples of pine gum.

Additional research on process and product development is in progress under contract at Cornell University, Ithaca, New York, on the synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers; and at the University of Florida, Gainesville, Florida, on the development of a practical process for the conversion of α-pinene to dimers in good yields, and the conversion of these dimers to useful, reactive derivatives.

Research in the field of chemical composition, physical properties and structure is in progress under a grant of P.L. 480 funds to the Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for development of new or improved methods of preparing selected terpene alcohols for use as standards, to obtain basic information on the composition and properties of products made from pine gum (project duration - 3 yrs.).

The Federal in-house scientific research effort in this area totals 10.0 scientific professional man-years. All of this effort is on process and product development. The contract research involves an additional 1.4 scientific man-years on process and product development. P.L. 480 research involves 1 grant for research in the field of chemical composition, physical properties, and structure.

The following lines of work under technology - process and product development - were terminated during the year: (1) Development of a method for the determination of rosin and rosin derivatives in protective coatings; (2) Application of the oxo and related reactions to terpenes and resin acids to produce alcohols, aldehydes, and/or acids and characterization of the products thus obtained; (3) Reaction of terpenes derived from turpentine with dienophiles to produce useful acids, aldehydes, amines, nitriles, sulfones, and related derivatives; (4) Development of practical methods for preparing the levopimaric acid-formaldehyde adduct, and the evaluation of this and selected derived products for industrial uses.

PROGRAM OF STATE EXPERIMENT STATIONS

One scientific man-year is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition, Physical Properties and Structure.

1. Composition and Physical Properties of Pine Gum. The contractor (Purdue Research Foundation) has developed additional information on the chemical transformations of terpene olefinic compounds by hydroboration and subsequent reactions. Thexyl(1,1,2-trimethyl propyl)borane was found to react stereospecifically with the endocyclic and exocyclic moities of d-limonene to produce only one diol isomer after oxidation and hydrolysis with alkaline hydrogen peroxide, whereas borane reacts indiscriminately with d-limonene to produce three diol isomers. A simple and convenient method was developed for the isomerization of Δ^3-carene to Δ^4-carene. Information obtained from

the selective hydroboration of terpenes such as d-limonene should provide the basis for the synthesis of potentially useful products from terpenes. This information should also be helpful in the synthesis of terminal difunctional terpene derivative that would be useful as synthetic intermediates for various types of industrial polymers or copolymers. Systematic studies of the selective hydroboration of model olefinic compounds and of terpene olefinic compounds containing various types of functional groups will be continued. (S4 1-112(C)).

Under a P.L. 480 project at the Patronato "Juan de la Cierva" School of Technical Investigations, Barcelona, Spain, research is being conducted to develop new or improved methods for synthesizing, isolating and purifying selected terpene alcohols for use as standards in obtaining information on the composition and properties of products made from pine gum. A considerable number of compounds of this type have been prepared, purified, and characterized, and details of their synthesis have been reported. Samples of 29 terpene alcohols together with spectral data of these and their intermediates have been provided. Samples of high purity terpene alcohols of known structure together with details of their synthesis and purification will provide useful information and standards for the instrumental examination of naval stores products incidental to their processing and their practical applications in industrial uses. (UR-E25-(50)-36).

B. Technology -- Process and Product Development

1. Development of Intermediates for the Production of Resins, Plastics, Plasticizers, and Other Industrial Products from Pine Gum and Its Components. The research to prepare polymerizable monomers for vinyl and condensation type polymers from terpene derivatives was discontinued and the effort expended on making polymerizable vinyl-type monomers from rosin. Vinyl tetrahydroabietate appears particularly promising. Higher yields of polymers are obtained with it than with the vinyl esters of commercial hydrogenated and disproportionated rosins. Homo- and copolymers having good molecular weights have been obtained. Copolymers with acrylonitrile, butadiene and styrene have given interesting polymer blends of the ABS type. Work was initiated to prepare vinyl ethers of commercial "Abitol" and tetrahydroabietol. Vinyl maleopimarate, and the acrylates of 2,2,3-trimethyl-3-cyclopentene-1-ethanol and the α-lactone of (6-hydroxy-1-methylethyl)-heptanoic acid, are other monomers from this research which have been used in polymer studies under project S4 1-89(C). (S5 2-55).

Research has been successfully completed on the reaction of terpenes derived from turpentine with dienophiles to produce useful acids, aldehydes, amines, nitriles and related products. The information obtained under this project goes a long way toward delineating the effect of the nature of the substituents on the double bond of the dienophile on its reactivity with α-terpinene and on the number and relative abundance of isomeric adducts. Reactant concentration, as well as reaction temperature, has been found to affect the product distribution. This information should be generally

useful in predicting the behavior of other 1 or 1,4 substituted cyclic dienes with dienophiles and may help clarify the precise mechanism of the Diels-Alder reaction. Many new reactive terpene derivatives have been made available for further evaluation. The α-terpinene adducts have been found to have somewhat hindered functional groups and are relatively stable to hydrolysis. Evidence was also obtained showing that the maleic and fumaric adducts behave abnormally. The fumaric adducts contain a third component, as yet unidentified. Esterification of the maleic anhydride adduct by means of methanol and tetramethyl ammonium hydroxide gives the dimethyl ester of the fumaric acid adduct. Suitable reaction conditions for preparation of adducts of alpha-terpinene with most of the more readily available dienophiles have been determined. In some cases these same products have been made from α-pinene by in situ isomerization. (Research to determine and optimize yields is still in progress). By these processes terpenyl acids, anhydrides, aldehydes and nitriles have been produced as primary products. Amines and alcohols can be made by reduction of the corresponding nitriles or aldehydes. The acrolein adduct is a particularly attractive raw material because it can be converted to the alcohol, the acid or amines as well as condensed with ketones. Another two-step process for converting α-pinene to secondary amines has also been developed. This consists of selective halogenation by t-butyl hypochlorite generated in situ followed by reaction of the halides with a primary amine. (S5 2-48).

Research has been started to produce useful chemicals from terpenes and resin acids by free radical addition of functional groups. Initial work has emphasized the development of rapid and reliable methods of analysis suitable for use in mixtures of resin acids or terpenes, peroxides, and halocarbons. Once analytical methods are established relative reactivities of the various terpenes and resin acids will be determined. (S5 2-57).

Practical processes have been developed for the preparation of the formaldehyde adduct of levopimaric acid from pure levopimaric acid, resin acid mixtures, and slash and longleaf pine gums, and for derived products such as an hydroxy acid, 6-hydroxymethyldihydroabietic acid, and an interesting glycol, 6-hydroxymethyltetrahydroabietinol. Considerable industrial interest has been shown in the products; several companies are evaluating them. Possible markets for the residual rosin include metal resinates, paper size, etc., where the combined formaldehyde would not have a deleterious effect and might give improved products. This research has been completed and the research effort redirected toward using rosin in polyurethanes. (S5 2-51).

Research has been initiated to develop economical processes for the preparation of polyglycols and polyethers from rosin, resin acids and pine gum for use in polyurethane elastomers and foams. Some of the new polymers derived from the vinyl ester of hydrogenated rosin such as the copolymers with vinyl chloride, with acrylonitrile and butadiene and with vinyl acetate, should have considerable industrial value. Vinyl ethers of hydrogenated rosin should also have value. Amides, amines and nitriles from rosin are easily prepared. Such derivatives from hydroxylated resin

acids will be new and their acrylates and vinyl ethers should give interesting polymers. The amino alcohols could also be of considerable interest. Oxonated rosin prepared from cobalt octacarbonyl is an attractive rosin derivative in that it can be converted by copper chromite reduction to a glycol, amino alcohol, etc. Glycol esters of rosin and rosin adducts are interesting materials and should find application in polyurethanes. Plans for future research involve the continuation of work outlined above. In addition, epoxidation of dehydroabietic acid contained in commercial hydrogenated rosin and the procedure for oxonating rosin will be investigated. (S5 2-56)

The contractor (University of Florida) has continued the investigation of the acid-catalyzed dimerization of α-pinene. The results obtained thus far demonstrate that several types of diterpenes can be prepared from α-pinene and that proper choice of conditions can give some measure of control over the product composition. Major progress has been made toward establishing the structures of several of the products and especially in eliminating some of the more obvious possibilities. The relatively good stability of some of the products suggests possible utility as high boiling solvents or as functional fluids. The production, separation, characterization and proof of structure of all the isomeric 3,3'-dimers of cis-pinane makes pinane dimers of precisely known structure available for the first time. The fact that all isomers can be prepared has fundamental significance regarding the steric requirements of the pinane ring. Immediate emphasis of future work will be on structural correlations and on ozonolysis. (S5 2-49(C)).

2. Conversion of Rosin Acids, Pine Gum and Turpentine into New Polymers, Protective Coatings, Resins and Plastics. Research has continued on the preparation and reactions of epoxides and ozonization products of resin acids and their derivatives. Several types of polyimide-polyamide polymers have been prepared from maleopimaric acid chloride and polyamines. Some of the polymers could be cold drawn from a hot melt to give fibers. When the diamine used was 1,6-hexanediamine, either alone or in conjunction with other aliphatic diamines, good films were produced. Some polymers had softening points as high as 350°C and were resistant to a wide range of solvents. However, films could not be obtained exhibiting the stability toward ultrahigh temperatures reported for films prepared from pyromellitic dianhydride. In other work, studies of reactions of levopimaric acid diepoxide indicate that this compound probably has the novel 1,4-2,3-diepoxide structure. A number of new derivatives of maleopimaric acid were prepared and characterized, for testing as agricultural chemicals. Several of these materials showed activity; two of them were active as nematocides at low concentrations. In addition, POPG and POPG-diepoxides also showed activity. The addition of dihalocarbene, formed in situ by the reaction of sodium hydroxide and chloroform, to gum rosin and to the methyl ester of rosin was carried out. GLC analysis showed that all the abietic, neoabietic, palustric, and isopimaric acid esters reacted and about 15% of the pimaric acid. Dehydroabietic acid did not react. The resulting product showed considerably enhanced stability toward air oxidation. If further tests show epoxidized ester gum to be competitive with epoxidized soya oil, as a

stabilizer for poly-(vinyl chloride), a large new outlet for gum rosin could be established. Several derivatives of maleopimaric acid as well as POPG and POPG-diepoxides show activity as agricultural chemicals and thus a potentially large new outlet for rosin exists in this area. The stabilization of rosin towards air oxidation by reaction with dihalocarbenes might open up new markets for gum rosin, e.g. in the field of hot melt adhesives. Research will be continued on the chemistry of resin acid epoxides. (S5 2-52).

Research to develop improved polyester resins from rosin derivatives has continued. Polyesters prepared from dimerized rosin acid were found to have poor compatibility with styrene, but it was possible to improve compatibility by treating the polyester reaction products with sufficient acetic anhydride to convert part of the rosin to the rosin anhydride. A simple, low-cost process for the preparation and isolation of dehydroabietic acid from gum rosin has been developed and could lead to expanded uses for this product. Several potentially useful derivatives of this acid are known but the difficulty of obtaining the acid in a reasonably pure state has discouraged development work on them. For example, amino-dehydroabietic which can be readily prepared should yield polyamide resins useful for modifying Nylon-type resins. The chloromethylation of dehydroabietic acid should be useful in the preparation of polyester resins. A practical, low-cost method was also developed for the preparation of the monoethylene and monopropylene glycol esters of rosin by direct reaction of the rosin with ethylene oxide and propylene oxide, respectively. These monoglycol esters will be useful in making polyesters, and should have potential for applications in adhesives, alkyd resins, and surface coatings. In addition, the free alcohol group of the monoglycol esters can be readily converted to the corresponding vinyl ether, giving valuable reactive sites. In connection with analysis of rosin samples for the research on polyesters, two new resin acids--sandaracopimaric acid and Δ^8-isopimaric acid--were discovered. Another phase of work has led to the development of a new and improved technique for the preparation of copper resinates from rosin or pine gum. Work will be continued on the direct condensation of ethylene and propylene oxide with rosin to yield monoglycol esters, and the utilization of these esters in polyester resins. (S5 2-53).

Contract research at the University of Cincinnati on the application of the oxo and related reactions to terpenes and resin acids to produce alcohols. aldehydes, and acids has been completed. The reaction of olefins with carbon monoxide has been applied to produce new primary alcohols and aldehydes but the yields were not as good as was hoped for and the production of glycols or dialdehydes was not very good. Conditions have been found for the preparation of 9-formyl-p-menthene-1 in 45-50 percent yield and of the methyl ether of 9-methylol-p-menthene-1 in about 60 percent yield. 1-Formyl(or methylol)-p-menthene has also been made in about 30-40 percent yield but an equal amount of nonvolatile byproduct was also formed. Although the aldehydes and primary alcohols produced in this research may be useful in preparing acids, esters and amines, the yields and

complexity of products from the oxo reaction makes it doubtful whether commercial production would be economical. (S5 2-45(C)).

In contract research at Cornell University to synthesize terpene alcohols and glycols by reaction of selected terpenes with formaldehyde yields of monohydric alcohols from both camphene and limonene were increased. Product purities were also improved. At the same time the processes were simplified. Using the improved procedures, good yields of a 1-hydroxymethyl-p-menthadiene were obtained from α-terpinene. However, α-pinene continued to give mediocre yields of mixed products. On the basis of yield, product purity, reaction conditions, and availability of starting material 8-hydroxymethyl camphene is the most promising product. It has been selected as one of the two alcohols to be converted to esters and further evaluation. Hydroxymethyl tricyclene and 9-hydroxymethyl limonene each has advantages and final selection of one for evaluation may depend on the ease with which their esters can be prepared. No diol was obtained in good enough yield for further evaluation. (S5 2-46(C)).

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AREA 9 - SWEET SORGHUM UTILIZATION - FOOD

Problem. The Lower Rio Grande Valley, which is largely dependent on an agricultural economy, must have a greater selection of crops for diversification to meet unfavorable environmental and marketing conditions that frequently beset the area. Freezes in 1949 and 1951 and again in 1962 destroyed many citrus groves and generally retarded citrus production--a valuable source of farm income. Cotton, a mainstay crop, is also a surplus crop. In addition, yields in the Valley are frequently low, and insects and root rot pose troublesome problems. Many vegetables do well, but heavy losses have resulted from freezes, heavy rains, and maturation times that place the Valley vegetables in direct market competition with those grown in other parts of the U. S.

Sweet sorghum has potential for becoming a profitable diversification crop. There are now available new disease-resistant varieties with high sugar content. This factor, together with favorable world sugar prices, has encouraged consideration of sweet sorghum canes as a potential sugar crop for the Valley. The modest water requirements of sorghum and the sub-tropical climatic conditions conducive to an extended growing season increase its attractiveness. In addition, preliminary studies in processing encourage evaluation of this crop for the production of sugar; its process integration with those for beet and sugarcane would extend the use of costly raw sugar installations.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving an organic chemist engaged at the U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas, in research to determine the chemical characteristics of juices obtained from selected new varieties of sweet sorghum canes grown in the Rio Grande Valley of Texas, and the effects of cultural and harvesting practices on the chemical characteristics in relation to suitability for sugar recovery; close cooperation is maintained with Substation 15, Texas Agricultural Experiment Station, Weslaco, Texas, for growing and harvesting breeding stock provided by the Crops Research Division.

The Federal scientific effort at the Southern Division devoted to research in this area totals 1.2 scientific man-years. All of this effort is on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 2.5 scientific man-years is devoted to sweet sorghum and sugar crops utilization research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Investigations of Chemical Characteristics of Sweet Sorghum to Evaluate Its Potential for Recovery of Sugar. Investigations of the chemical characteristics of new sweet sorghum canes to determine their suitability for sugar recovery have continued in cooperation with the Texas Agricultural Experiment Station at Weslaco and with the Crops Research Division. Such research on sweet sorghum canes in 1964 contributed to the selection and reevaluation of eleven nursery lines and varieties for 1965 planting tests. Analyses of the 1965 field test samples of sorghums from four areas are nearing completion. These data are essential in the evaluation of varieties and cultural practices. Simultaneously with these routine analyses, limited juice clarification studies were made, and means found to obtain flocculation and precipitation of more than 95% of the starch in the raw juice without use of heat. There have not yet been enough tests to establish the exact conditions under which optimum eliminations of starch, protein, bagacillo, and other objectionable impurities are attained, but a pH range of 7.5-8.5 appears to be important, as well as a juice Brix of 12-14% and the use of some polyelectrolyte such as Separan AP-30 at the level of 1-5 ppm. Evaluation of these extensive data should reasonably well establish the potential for growing in the Rio Grande Valley varieties with good prospects for commercial sugar production; however, progress has been limited in developing methods for purification of the juice for sugar recovery. (S5 5-51).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

None.

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Color, Texture and Other Quality Factors

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Technology--Process and Product Development

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AREA 10 - RICE UTILIZATION - FOOD

Problem. The productive capacity of U. S. rice growers has increased faster than domestic and export consumption over the past decade, thus limiting the income potentially available from this major world food grain. Detailed knowledge of chemical composition and physical properties as related to processing is needed to guide milling, processing, and product development of U. S. rices so that they can better meet the quality and new product requirements needed for expanded markets. New and diverse food products that are economical to manufacture, convenient to prepare, and attractive in flavor and texture are needed to increase the total consumption of rice both domestically and abroad. Additional needs include the development of improved milling machinery and techniques, primarily to increase the yield of head rice; intensified research on deep milling to evaluate and utilize the protein flour and residual kernels produced by this technique; and research to provide greater flexibility in the industry by developing from either medium or long-grain rice new products that can be cooked to provide either discrete kernels or a gelatinous food.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program at New Orleans, Louisiana, involving biochemists and analytical chemists engaged in research on the chemical composition and physical properties of rice and its products. Distribution of the chemical constituents of milled rice in consecutive layers of the kernel is being studied, with special emphasis on nutritionally important constituents such as proteins, amino acids, starch, lipids, vitamins, and minerals; and on the cooking and chemical characteristics of the kernels remaining after differential removal of these layers. Findings from this research will provide the necessary basis for evaluating the economic feasibility of using high-protein rice flours (layers removed by deep milling) for protein fortification of foods and as dietetic or other specialty foods. Close cooperation is maintained, under formal memoranda of understanding, with the Louisiana, Arkansas, and Texas Rice Experiment Stations, who supply rice samples of known variety and cultural history for the experimental studies. The Rice Inspection Service, Grain Division, AMS, New Orleans, Louisiana, cooperates by providing assistance in grading rice samples from the research investigations. Cooperation is also maintained with the Western Division.

Other research on chemical composition and physical properties has been initiated under a P. L. 480 grant to Kyoto University, Kyoto, Japan. Scientists at this institution will study the distribution of the major proteins of rice within subcellular particles and the distribution of these particles in the cellular structure of the rice kernel to obtain basic information needed to develop new and improved rice products and methods of producing them (project duration--3 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 1.7 scientific man-years, all of which are presently applied to chemical composition and physical properties.

Under P. L. 480 research there is presently one grant, also on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 3.0 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Investigation of the Distribution of Chemical Constituents in the Rice Kernel. The physical properties of high-protein rice flours, which were prepared by tangentially abrading successive layers from conventionally milled Bluebonnet-50 long-grain rice, were investigated. Determination of the hot paste viscometric properties and pasting characteristics of the eight successive flour layers (each representing about 6-7% by weight of the original milled rice) showed that none of the layers exhibited a peak or set-back when the rice paste was heated to and above the gelatinization temperature. In contrast, the hot paste viscosity of the original milled rice peaked at 1250 Brabender Units (BU). The residual kernels obtained after removal of each of the layers exhibited successive increases in peak viscosity--1450 to 1800 BU--from the first to the sixth layer. An unexplained decrease in peak viscosity--1660 to 1600 BU--was shown by the kernels remaining after the removal of the seventh and eighth layers. The viscometric properties of these high-protein rice flours are quite unique and different from those of any commercial rice flour, and must be taken into consideration in the development of new uses for these flours.

The chemical composition and viscometric properties of flours prepared from whole, second heads, and parboiled rice in a commercial Satake mill were investigated to determine feasibility of adapting commercial equipment to high-protein flour preparation. One and a quarter tons of Belle Patna rice (7.3% protein, 0.9% fat, 91.7% starch) was abrasively milled and five successive fractions separately collected for a total of 14.4% wt. removal. Flour:chits ratio (80-mesh) was approximately 60:40 except for 40:60 in fraction 1. Protein in flour progressively decreased from 15.1 to 14.5%; fat 12.8 to 1.7%; and starch increased 42.9 to 79.1% in successive fractions. Values of the chits were: 11.7 to 8.5% protein, 6.1 to 0.3% fat, and 65.5 to 89% starch. Brabender viscograms of the 5 flours indicated unusually low values for the pasting characteristics with a slight progressive increase from fractions 1 to 5. Residual kernels exhibited progressively higher values of pasting characteristics than did the initial feed. Chits were intermediate in values, tending to approach those of the residual

kernels. Similar analyses were conducted on high-protein flour prepared by removal of two successive fractions from Belle Patna second heads and from parboiled rice. In addition, two 200-lb. samples of flour were commercially prepared from head and second head Belle Patna for sample distribution. This evaluation of flours prepared with the Satake mill demonstrates that it may be possible to adapt commercial equipment to high-protein rice flour preparation. The development of the product can be a useful adjunct to the domestic rice milling and reprocessing industries in that it provides a whiter rice with improved cooking characteristics and a high-protein flour that may be useful in many domestic dietary products. Since the residual kernels are high in starch and low in protein and fat, they should also provide more uniform stock for industrial uses. (S1 4-13).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

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Technology--Process and Product Development

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Color, Texture and Other Quality Factors

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AREA 11 - DECIDUOUS FRUIT AND TREE NUT UTILIZATION - FOOD

Problem. The peach industry in the Southeastern United States is dependent to a large extent on the fresh market. For example, 19,405,000 bushels of peaches were produced in the South Atlantic and South Central States in 1965; of this total, 13,188,000 bushels from both areas were sold on the fresh market and only 2,846,000 bushels--none of which were grown in the South Central States--were processed. A peach processing industry is needed in the Southeastern States to provide a profitable market for more of the edible peaches that do not meet fresh market standards and to rapidly convert a higher proportion of the overall crop to stable forms. Basic information, not now available, on the flavor components of peaches is needed to guide development of improved processed products from southern grown fruit.

Climatic conditions that favor rapid deterioration of fresh peaches both on and off the tree, erratic ripening periods and markets, and short-lived peach orchards are other factors contributing to the need for more extensively integrated fresh market-processing operations. Technical problems preventing the more rapid development of the peach processing industry in the Southeastern States must be overcome. Many of the peach varieties grown in the Southeast require a modification of processing procedures to make satisfactory standard-type products. Still other varieties cannot be used in standard-type products, and new food forms must be found for them. Recent rapid advances in food science and processing technology make it possible through research to develop both new and improved peach products. These are needed to bolster the economics of the South's peach industry, as well as to provide the superior qualities and greater convenience in food products, which the consumer now demands.

USDA AND COOPERATIVE PROGRAM

The Department has a program of basic research on peaches being conducted under contract by the Georgia Agricultural Experiment Station, University of Georgia, Experiment, Georgia. Food chemists and food technologists conduct this research. Research to develop basic information on the flavor of peaches, particularly varieties grown in the Southeastern States, is in progress under one contract. Specifically, the objective of this research is to isolate, identify, and characterize the constituents of peach flavor and aroma, and to acquire information needed to guide development of improved processed products from the fruit.

The contract research involves a total level of effort of 0.5 scientific man-years, all of which is devoted to an investigation of flavor.

One line of work (under Technology--Process and Product Development) was discontinued during the reporting year. Conducted by the same contractor,

Georgia Agricultural Experiment Station, it concerned research on optimum procedures for the production and preservation of puree and clear juice peach concentrates, for the preparation and the handling under simulated commercial conditions of refrigerated fresh peach slices, and for canning Southeastern peaches; and on the development of partially dehydrated pasteurized peach products. This research was carried out with the support of the Area Redevelopment Authority of the Department of Commerce.

PROGRAM OF STATE EXPERIMENT STATIONS

A total of 50.0 scientific man-years is devoted to this area of research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Flavor

1. Basic Studies on Flavor and Odor Constituents of Peaches. Analysis of the flavor and aroma constituents of Southeastern peaches is being conducted under contract to the Georgia Agricultural Experiment Station. Gas chromatographic procedures were used to analyze the sugars (trimethylsilyl derivatives) from Coronet, Southland, and Sullivan Elberta peaches at three stages of ripeness. It appeared that sucrose was predominant (57-74%) in all three varieties and at all different stages of ripeness. In general, fructose and glucose were comparable except for Sullivan Elberta peaches, in which fructose increased through the shipping ripe, firm ripe, and soft ripe stages, while glucose, in contrast, decreased. For Coronet peaches ripened off the tree, great decreases in mannose and fructose were found. Other compounds noted were galactose and sorbitol. Studies are continuing to identify the volatile constituents present in an oily residue isolated from fresh peaches. Among compounds tested, gamma decalactone, ethyl heptylate, ethyl acetate, and ethyl acetoacetate agree in retention time with peaks from the peach oil, but more work is needed to positively establish their presence. The oil residue of fresh Sullivan Elberta gave different peak areas according to the degree of ripeness. The most significant change was the increase of peak area in the compounds with a long retention time in firm ripe as compared to shipping ripe. These observations together with others may indicate that as the peaches become riper the amount of high molecular weight compounds increases. (S3 2-44(C)).

B. Technology--Process and Product Development

1. Development of New and Improved Processed Products from Southeastern Peaches. Valuable technical information on the development of new and improved processed products from Southeastern peaches has been obtained in recently completed contract research conducted by the Georgia Agricultural Experiment Station. Peach puree. A process has been developed for the manufacture of a puree-type peach drink of twofold strength, and for

the bulk manufacture of peach puree vase for drinks and "concentrates." Clear peach juice and concentrate. A process has been improved for the manufacture of a clear peach juice and concentrate. This product can profitably utilize the 10% to 25% of the total production of peaches that fails to meet standards for shipping fresh, canning, pickling, or use in peach puree products. Refrigerated fresh peach slices. A process has been improved for the manufacture of refrigerated fresh peach slices without pasteurization treatment. Canned peaches. The extensive data obtained will be helpful in raising the U. S. Standard Grade of canned Southeastern peaches. These data include effects of early-, mid-, and late-season varieties, stage of maturity, preprocessing treatment, canning medium, and the like. Dehydrated peach flakes. Limited samples of the product developed in cooperation with SU have been prepared for evaluation and distribution. Other. Additional progress achieved under this contract includes a demonstration of (1) the potential of the gas chromatographic profile for ready detection of quality differences in peach products, and (2) the wide variance of total tannins, leucoanthocyanins, and flavonols in different varieties, and their gradual increase as the fruit matured. These products and results should augment the utilization of Southeastern peaches. (SU-0-0-1 (DC)).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

None

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Line Project Check List - Reporting Year July 1, 1965, to June 30, 1966

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S1 4-	Rice Utilization Investigations-Southern Region			
S1 4-13	A study of the distribution of the constituents of rice in consecutive layers of the kernel and of the evaluation of selected fractions and degrees of milling, as a basis for the development of new and/or improved rice products.	New Orleans, La.	Yes	10-A-1
S2 1-	Cotton Utilization Investigations			
S2 1-183 (C)	Investigation of the effects of mechanical treatments prior to, during, and following resin finishing on the ease-of-care properties of fabrics and garments.**	Raleigh, N. C.	Yes	1-C-3
S2 1-195	Investigation of radiochemical yields of high-energy			
S2 1-195 (Rev.)	radiation activated reactions of cotton to develop improved cotton products.** *	New Orleans, La.	Yes	1-B-2
S2 1-197 (C)(Rev.)	Evaluation of stretch-type cotton yarns in knit wear.	Raleigh, N. C.	Yes	1-C-8
S2 1-200 (C)(Rev.)	Development of weather-resistant, water-repellent finishes for cotton.	Denton, Texas	Yes	1-C-4
S2 1-2-4 (C)	The aerodynamic separation of lint cotton into individualized fibers to provide information needed for improving cotton textile processing equipment.**	Westbury, L. I., N. Y.	Yes	1-C-2
S2 1-205 (C)	The development of cotton knit fabric having increased bulk, warmth, and dimensional stability.**	Clemson, S. C.	Yes	1-C-8
S2 1-206 (C)	A determination of the structural components of the cotton fiber that contribute most to tensile strength and how they can be utilized to increase tensile and recovery properties to produce cotton products having enhanced physical properties.**	South Pasadena, Calif.	Yes	1-A-2
S2 1-207	Development of guides for the maximum utilization of cottons of varying fiber properties.	New Orleans, La.	Yes	1-B-5
S2 1-208	Investigation of the effects of gross and fine			
S2 1-208 (Rev.)	structures of the cotton fibers on their physical behaviors.** *	New Orleans, La.	Yes	1-A-2
S2 1-209 (Rev.)	Microscopical investigations of absorption phenomena in native, mercerized, and modified cottons.	New Orleans, La.	Yes	1-A-1
S2 1-214	Separation and identification of the cleavage products			
S2 1-214 (Rev.)	of partially etherified cottons, including cross-linked cottons.** *	New Orleans, La.	Yes	1-A-4
S2 1-216	A study of reactions between epoxy compounds or their halohydrin precursors and cotton cellulose.**	New Orleans, La.	Yes	1-B-1
S2 1-217 (C)	Effect of variation in structure on cotton fiber properties caused by environmental and genetic factors to obtain basic information important in optimum utilization of cotton.	College Station, Texas	Yes	1-A-2
S2 1-219	Improved methods of etherifying cotton cellulose.**	New Orleans, La.	Yes	1-B-1
S2 1-221 (C)	Investigations to determine the effects of fiber extensibility on fiber breakage in mechanical processing.	Knoxville, Tenn.	Yes	1-A-3
S2 1-222 (C)	Treatment of cotton fibers by sonic energy to obtain basic information required for the development of improved equipment for processing cotton into textiles.	Carteret, N. J.	Yes	1-B-5
S2 1-223 (C)	Effect of the soiling environment on the soiling tendency of a series of cotton finishes.	Washington, D. C.	Yes	1-B-4

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S2 1-224 (C)	Determination of optimum yarn constructions, knitting structures and prefabrication design for producing stretchable articles of knitted cotton wearing apparel by slack mercerization.	Raleigh, N. C.	Yes	1-C-8
S2 1-225 (C)	The relationship of molecular size, nature, shape, conformation, and configuration of organic non-aqueous compounds to their swelling power on cotton cellulose.	Brooklyn, N. Y.	Yes	1-A-1
S2 1-226	The development of cotton fabrics having improved warp and filling stretch properties by a comprehensive investigation of fabric and yarn structures and processing conditions during slack mercerization and resin treatment.**	New Orleans, La.	Yes	1-C-8
S2 1-227	Excellent smooth drying and wrinkle resistant fabrics by crosslinking cotton with highly reactive methylolamide amino acid derivatives.**	New Orleans, La.	Yes	1-C-3
S2 1-228 (C)	Investigation of the physics of seam pucker in relation to fabric structure to develop improved sewing thread for wash-wear cotton products.	Atlanta, Ga.	Yes	1-C-3
S2 1-229 (C)	Development of a method for counting neps in cotton at various stages of textile processing.	South Pasadena, Calif.	Yes	1-A-7
S2 1-231 (C)	An investigation of the chemical modification of cotton through treatments with reagents in the vapor phase.	East Greenwich, R. I.	Yes	1-B-1
S2 1-232	The preparation of new finishing agents for cotton and cotton derivatives based upon lead and other metal compounds.**	New Orleans, La.	Yes	1-B-1
S2 1-233	Preparation of fatty acid or hindered acid esters of cotton to form new or improved end-use textile products and investigation of reaction mechanisms involved.**	New Orleans, La.	Yes	1-B-3
S2 1-234	Investigation of blending methodology to establish optimum blending procedures for maximum utilization of cottons differing widely in fiber properties.**	New Orleans, La.	Yes	1-C-1
S2 1-235	Improvement of smooth drying properties, wet crease recovery, and moisture absorptivity in wash-wear cotton through swelling treatments.**	New Orleans, La.	Yes	1-C-3
S2 1-237 (Gr)	Investigation of the configurational interactions between fibers and yarns in the region of local deformations in woven cotton cloth.	Cambridge, Mass.	Yes	1-A-5
S2 1-238 (Gr)	Correlation of surface microtopography of treated and untreated cotton fibers with resistance to soiling of cotton textiles.	Tucson, Ariz.	Yes	1-B-4
S2 1-239 (C)	Development of wash-wear cotton fabric with improved moisture absorptivity by use of reactive swelling agents.	Birmingham, Ala.	Yes	1-B-1
S2 1-240 (Gr)	An exploratory study of the crosslinking of chemically modified cotton to obtain cotton fabrics with an optimum combination of resiliency and thermoplasticity.	Princeton, N. J.	Yes	1-B-1
S2 1-241 (C)	Investigation of factors influencing comfort in cotton apparel fabrics.	Washington, D. C.	Yes	1-A-6
S2 1-242 (C)	To determine optimum processing procedures for cotton differing in tensile and elastic properties and relate these properties to mechanical processing performance, yarn, and fabric properties.	Auburn, Ala.	Yes	1-C-1
S2 1-243 (C)	Development of finishes for cotton fabrics to render them more rapid drying.	Washington, D. C.	Yes	1-B-1
S2 1-244 (C)	Development of improved coated cotton fabrics with optimum strength-weight characteristics for outdoor uses.*	Dedham, Mass.	Yes	1-C-4

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S2 1-245 (C)	The development of weather resistant cotton textiles with improved physical properties by interfacial and graft polymerization.	Birmingham, Ala.	Yes	1-C-4
S2 1-247	An investigation of the chemical modification of cotton fabrics using reagents in the form of fogs or aerosols.	New Orleans, La.	Yes	1-B-1
S2 1-248 (Gr)	Elucidation of the role of fiber morphology on frictional behavior important in mechanical processing of cotton fibers and in the behaviors of cotton products.	Atlanta, Ga.	Yes	1-A-5
S2 1-249	Investigations of cotton fiber-property changes during mechanical and chemical processes, which are responsible for altered sorption of alkali solution.	New Orleans, La.	Yes	1-A-1
S2 1-250	Treatment of cotton with fluorochemicals to produce a finish with low surface energy.**	New Orleans, La.	Yes	1-C-5
S2 1-251	Exploratory research to impart multifunctional properties to cotton through the use of specially tailored compounds.**	New Orleans, La.	Yes	1-C-6
S2 1-252	Development and evaluation of a new machine for opening and blending bales of cotton in any desired proportion in textile mills.	New Orleans, La.	Yes	1-C-2
S2 1-253	Wash-wear cottons of high abrasion resistance by the application of durable polymeric coatings.**	New Orleans, La.	Yes	1-C-3
S2 1-254	Development of optimal cotton fabric structures for men's trousers and dress suits.	New Orleans, La.	Yes	1-C-3
S2 1-255	Investigation of effective crosslinks in cotton modified by chemical treatment.**	New Orleans, La.	Yes	1-A-4
S2 1-256	Development of multipurpose finishes for outdoor cotton fabrics with improved physical properties.	New Orleans, La.	Yes	1-C-4
S2 1-257	Development of durable inexpensive flame retardants for cotton.**	New Orleans, La.	Yes	1-C-6
S2 1-258	Exploratory investigation of reversible chemical reactions to obtain information basic to the development of a commercially feasible reversible crosslink.	New Orleans, La.	Yes	1-B-1
S2 1-259	The fixation of antimicrobial agents in cotton fabric by use of zirconium compounds, to impart improved weather resistance.	New Orleans, La.	Yes	1-C-4
S2 1-260	Investigation of resistance to edge abrasion in wash-wear cotton and methods for improvement.	New Orleans, La.	Yes	1-B-1
S2 1-261	Investigation of spatial and structural effects of reversible and conventional crosslinks in cotton.	New Orleans, La.	Yes	1-B-1
S2 1-262	An investigation of tensile and torsional or bending recoveries of single fibers, yarns, and fabrics of wash-wear treated cottons under wet and dry conditions.**	New Orleans, La.	Yes	1-B-5
S2 1-263	Microscopical investigations of chemical substitution and crosslink formation in cotton, to provide information basic to research required for increased utilization of cotton.	New Orleans, La.	Yes	1-A-2
S2 1-264	Investigation of the fluorescence spectra of native and modified cottons, to obtain information needed in the development of improved textile products.	New Orleans, La.	Yes	1-A-4
S2 1-266 (C)	Development of a research instrument for accurately and automatically determining length, length distribution and diameter of cotton fibers.	South Pasadena, Calif.	Yes	1-A-7
S2 1-267	Wash-wear fabrics of increased strength, durability, and luster by crosslinking fabrics woven of premercerized yarns.	New Orleans, La.	Yes	1-C-7

** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S2 1-268	Development of new basic information concerning the reactions of cellulose by use of nuclear magnetic resonance and other spectroscopic techniques to facilitate research in the chemical modification of cotton.	New Orleans, La.	Yes	1-A-4
S2 1-269	Investigations to improve the production and the performance characteristics of chemically treated cotton batting.	New Orleans, La.	Yes	1-C-8
S2 1-270	Investigation of the formation of free radicals in fibrous cotton cellulose and the reaction mechanisms of the activated cellulose with selected reagents, to develop new and improved cotton products.	New Orleans, La.	Yes	1-B-2
S2 1-271	Development of improved insect-proof cotton bags for the storage and shipment of food commodities, to maintain and expand markets for cotton.	New Orleans, La.	Yes	1-C-9
S2 1-272	The relation of fiber properties to fabric behavior in chemically treated cotton fabrics.	New Orleans, La.	Yes	1-B-5
S2 1-273	Development of a method for removing short fibers and improving fiber parallelization at textile carding machines.	New Orleans, La.	Yes	1-C-2
S2 1-274	The effect of high production carding on fiber length distribution and formation of fiber hooks in cotton.	New Orleans, La.	Yes	1-C-1
S2 1-275	Development of more reliable methods of appraising damage done by abrasive action on all-cotton wash-wear fabrics.	New Orleans, La.	Yes	1-A-7
S2 1-276	Investigation of new and improved x-ray diffraction techniques for the study of the crystalline structure of cotton and chemically modified cotton in contact with various interacting liquids.	New Orleans, La.	Yes	1-A-4
S2 1-277	Investigation of the chemical kinetics of cellulose etherifications to expand utilization of cotton.	New Orleans, La.	Yes	1-B-3
S2 1-278	Fundamental investigation of basic actions in cotton textile processing by means of high speed photography.	New Orleans, La.	Yes	1-C-2
S2 1-279	Development and evaluation of prototype equipment for feeding cotton to textile cards to produce cotton products with improved physical properties.	New Orleans, La.	Yes	1-C-2
S2 1-280	Investigation of accessibility to complexing agents of stable cotton cellulose derivatives.*	New Orleans, La.	Yes	1-A-4
S2 1-281	New and modified carbamate finishes for deferred cure processing to yield chlorine-resistant, lightfast cotton fabrics.*	New Orleans, La.	Yes	1-C-3
S2 1-282	Investigations of reactions between cotton cellulose and heterocyclic compounds to develop improved textile products.*	New Orleans, La.	Yes	1-B-1
S2 1-283	Investigation of new ethers and thioethers of cotton cellulose to develop new commercially attractive textile products.*	New Orleans, La.	Yes	1-B-1
S2 1-284	Investigation of plastic and oriented states of cotton cellulose to develop permanently shaped wash-wear textiles of long wear life.*	New Orleans, La.	Yes	1-A-2
S2 1-285	An investigation of the tensile recovery behavior of chemically modified cotton yarns and fabrics to facilitate the development of improved cotton products.*	New Orleans, La.	Yes	1-B-5
S2 1-286	A study of the blending of treated and untreated cotton fibers as a means of improving properties such as resistance to abrasion in wash-wear apparel thus permitting all-cotton to compete successfully with cotton synthetic blends.*	New Orleans, La.	Yes	1-C-3

* Initiated during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S2 1-287	A spectroscopic investigation of the molecular changes in structure occurring during the chemical modification of cotton cellulose to facilitate the development of new and improved textile products.*	New Orleans, La.	Yes	1-A-4
S2 1-288	Improvement of the abrasion resistance of durably creased cotton stretch fabrics and cotton fabrics by preferential crosslinking and polymer deposition treatments.*	New Orleans, La.	Yes	1-C-3
S2 1-289	Stable crosslinking agents suitable for use in delayed-cure processes for cotton.*	New Orleans, La.	Yes	1-C-3
S2 1-290 (C)	Investigation of the effect of resin thermoplasticity or thermosettability on the resistance of treated cotton fabrics to abrasion.*	Madison, Wis.	No	
S2 1-291	Development of basic information on the relationship between the spectral properties of selected chemicals and their suitability for improving cotton's properties.*	New Orleans, La.	Yes	1-A-4
S2 1-292	Treatment of cotton with finishes containing selected lead and other metal compounds to impart specific end use properties.*	New Orleans, La.	Yes	1-B-1
S2 1-294	Study of mechanisms involved in producing dry and wet crease resistant cottons by esterification with derivatives of monobasic acids.*	New Orleans, La.	Yes	1-B-3
S2 1-295	To determine the interaction of processing variables with yarn properties and end breakage in spinning at high processing speeds to produce cotton products of required properties at lowest cost.*	New Orleans, La.	Yes	1-C-1
S2 1-296	Improved abrasion resistance in cotton fabrics from crosslinking of partially swollen cotton.*	New Orleans, La.	No	
S3 2-	Citrus and Other Fruit Utilization Investigations - Southern Region			
S3 2-39 (C)(Rev.)	Investigation of the effect of maturity of grapefruit on total flavonoids, naringin, and poncirin; and on the chemistry, and nature of naringin and naringin-derived compounds to provide a scientific basis for the control of bitterness in processed grapefruit products.**	Norman, Okla.	Yes	6-A-2
S3 2-43	Investigations on conditions for drying as related to the storage stability and quality of "foam-mat" dried citrus products.	Winter Haven,Fla.	Yes	6-B-1
S3 2-44 (C)	Composition of flavor components of peaches (with emphasis on existing commercial varieties in the Southeastern United States).	Experiment and Athens, Ga.	Yes	11-A-1
S3 2-46 (C)	Development of practical and efficient pilot plant process for the manufacture of enzymatically debittered grapefruit juice and products with improved flavor, product stability, and storage characteristics.	Lake Alfred,Fla.	Yes	6-B-2
S3 2-47	Identification of recently isolated flavones and other neutral orange peel constituents and evaluation of their relation to bitterness and harshness in orange products.	Winter Haven,Fla.	Yes	6-A-2
S3 2-48	Study of the composition of essential oils in citrus products, particularly orange, to provide a basis for improvement in quality and uniformity of citrus products.	Winter Haven,Fla.	Yes	6-A-1
S3 2-49	Processed grapefruit products of greater attractiveness to the consumer through exploration of means to prevent or minimize the formation of bitter components in the fruit.	Winter Haven,Fla.	Yes	6-A-2

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S3 2-50	A study of off-flavor development in processed citrus juice in relation to the lipid composition of the suspended matter.	Winter Haven,Fla.	No	
S3 2-51	Influence of seasonal variations in color and flavor of Texas colored grapefruit on the quality of processed products made therefrom.*	Weslaco, Texas	Yes	6-A-2
S3 5-	Sweetpotatoes, Cucumbers, and Other Vegetable Utilization Investigations - Southern Region			
S3 5-22	Investigation to develop new and improved processed products from Southern-grown vegetables other than sweetpotatoes and celery, including cooperative studies with federal, state, and industry agencies.**	Weslaco, Texas and Raleigh, N. C.	Yes	7-C-1
S3 5-23 (Rev.)	Application of new basic information on the chemical constituents of celery stalk (petiole) to the development of processed products of improved flavor and convenience.	Winter Haven,Fla.	Yes	7-C-3
S3 5-24 (Gr)	Elucidation of molecular structure and chemical characteristics of the pectinase inhibitor in sericea forage and other plant sources.	Durham, N. C.	Yes	7-A-1
S3 5-25	Development of processing innovations for manufacture of stable, precooked, dehydrated sweetpotato flakes from roots of different varieties and environmental history.**	New Orleans, La.	Yes	7-C-2
S3 5-27	Adaptation of laboratory pure culture fermentation procedures to a commercially feasible process for the manufacture of pickled vegetable products.	Raleigh, N. C.	Yes	7-C-1
S3 5-28	Investigations of the chemistry and biochemistry of the carotenoid pigments in fruits and vegetables to facilitate the development of improved and more attractive processed products.	Raleigh, N. C.	Yes	7-A-1
S3 5-29	Investigation of effects of cucumber substrate, bacterial species and other environmental factors on the flavor and aroma of cucumbers and fermented cucumber products.	Raleigh, N. C.	Yes	7-B-1
S3 5-30	Application of innovations in food technology to the development of improved products from selected Southern-grown vegetables.*	Weslaco, Texas and Raleigh, N. C.	No	
S4 1-	Cottonseed, Peanut and Other Oilseed Investigations - Southern Region			
S4 1-89 (C)	Polymerization of vegetable oil, pine gum, and sugarcane derivatives and evaluation of properties of the polymers for use as elastomers, plastics, thickening agents, and protective coatings.**	Tucson, Ariz.	No	
S4 1-103 (C)	Investigations of gossypol esters and of mild oxidation products of gossypol and gossypol derivatives to develop information needed to aid the production of cottonseed meals and oils of the highest quality.**	Knoxville, Tenn.	Yes	2-A-2
S4 1-106 (C)	Investigation of the flavor and aroma components in processed peanut products.**	New York, N. Y.	Yes	5-B-1
S4 1-107 (C)	Chemical investigations of cyclopropenoids to develop means of eliminating or physiologically inactivating the cyclopropenoids found in cottonseed products.**	Urbana, Ill.	Yes	2-D-1
S4 1-109 (Rev.)	Investigations of the phospholipid, plasmalogen and other lipid or lipid-soluble constituents of peanuts and processed peanut products to expand the utilization of food grade peanuts.	New Orleans,La.	Yes	5-B-1

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S4 1-112 (C)	Investigations of chemical transformations of fat and terpene olefinic compounds by hydroboration and suitable subsequent reactions to produce useful products.	Lafayette, Ind.	Yes	2-A-3 8-A-1
S4 1-114	Development of methods for upgrading the quality of cottonseed oil by improving the color and eliminating undesirable components such as cyclopropene acids.**	New Orleans, La.	Yes	2-B-1
S4 1-115 (C)	Ethylene copolymerization with unsaturated fatty acid and gum naval stores compounds to extend the industrial utilization of agricultural products in commercial plastics.**	Tuscola, Ill.	Yes	4-B-1
S4 1-116	Isolation, identification, evaluation, and control of fungi and toxic fungal metabolites which may develop during processing of cottonseed and peanuts to improve the acceptance of their processed products.	New Orleans, La.	Yes	2-C-1 5-C-1
S4 1-117 (C)	Development of practical processing methods for inactivation of cyclopropene groups in cottonseed meal that decrease its value as a feed for laying hens.	Chicago, Ill.	Yes	3-C-1
S4 1-118 (C)	Development of peanut products for use in preparation and fortification of processed and convenience foods to extend usefulness of peanuts.	Auburn, Ala.	Yes	5-D-2
S4 1-119 (C)	A study of the relation of the carbohydrate, amino acid and protein components of the peanut to the formation of flavor and aroma during roasting, with the objective of expanding the direct utilization of this commodity.	Stillwater, Okla.	Yes	5-B-1
S4 1-120 (C)	Development of processing methods using peanuts of known history with respect to different growing, harvesting, and curing conditions that will provide processed peanut products of high quality and free of mycotoxins.	College Station, Texas	Yes	5-C-1
S4 1-121 (C)	Study of the limiting environmental conditions for the elaboration of mycotoxins in peanuts to develop information needed to assure the processing of highest quality peanuts.	Auburn, Ala.	Yes	5-C-1
S4 1-123	Study of rates of extraction of cottonseed with acetone-hexane-water solvent mixtures and nature and quantities of constituents in miscellas and rheological properties of marcs, to develop information basic to the production of cottonseed meal and oil of the highest quality.**	New Orleans, La.	Yes	3-C-1
S4 1-124	Preparation and evaluation of N-disubstituted fatty amides considered potentially useful as plasticizers, nitrile rubber softeners, and antifungal agents, to develop information basic to the increased utilization of cottonseed and other seed oils assigned to SU.	New Orleans, La.	Yes	4-B-1
S4 1-125	Improvement of processes for making cocoa butter-like fats from cottonseed and peanut oils and the development of data and processing techniques for improving the performance of confectionery fats.	New Orleans, La.	Yes	2-D-1
S4 1-126	Development of low-fat peanuts having acceptable peanut flavor and texture characteristics.	New Orleans, La.	Yes	5-D-2
S4 1-127	Investigation to determine processing conditions for the production of oils and meals of maximum quality from glandless cottonseed.	New Orleans, La.	Yes	3-C-1
S4 1-128	Development of new and improved techniques for preparing useful derivatives of cottonseed and peanut oils by esterification and interesterification.	New Orleans, La.	Yes	2-D-1

** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S4 1-129	Investigation of methods for correlating and predicting solubilities of homologous and analogous long chain saturated and unsaturated fatty acid derivatives.	New Orleans, La.	Yes	2-A-3
S4 1-130	Investigation of the chemical composition and characteristics of the protein systems of cottonseed to serve as a basis for improvement of nutritive value of cottonseed meal and flour.	New Orleans, La.	Yes	3-A-2
S4 1-132 (C)	Study of sterilizing or inactivating treatments in conjunction with artificial drying and curing of peanuts to develop processing conditions needed for producing mycotoxin-free roasted peanut products of optimum quality.	Stillwater,Okla.	Yes	5-D-3
S4 1-133	Inactivation or removal of aflatoxins from contaminated cottonseed, peanuts, and their products to permit utilization in foods and feeds.*	New Orleans, La.	Yes	3-B-1 5-C-1
S4 1-134 (Gr)	The development of procedures for synthesizing labeled malvalic acid esters.	Boston, Mass.	Yes	2-A-3
S4 1-135	Isolation of the cyclopropenoid fatty acid in cottonseed and cottonseed oil and the investigation of those chemical and physical properties of the cyclopropenoids important to the preparation and use of cottonseed products.*	New Orleans, La.	Yes	2-A-3
S4 1-136	Isolation and identification of cottonseed constituents that cause mortalities among swine.*	New Orleans, La.	Yes	3-B-1
S4 1-137 (C)	Biological studies of cyclopropenoid derivatives and cottonseed oils treated to remove cyclopropenoids to assure the production of wholesome commercial cottonseed oils.*	St. Louis, Mo.	No	
S4 1-138 (C)	Isolation and identification of the reaction products of gossypol with simple esters and model triglycerides.*	Savannah, Ga.	No	
S4 1-139	Development of suitable processes and processing conditions for inactivation of aflatoxin in cottonseed and peanut products using basic nitrogen compounds.*	New Orleans, La.	No	
S5 2-	Naval Stores Investigations - Southern Region			
S5 2-39 (Rev.)	Development of a method for the determination of rosin and rosin derivatives in protective coatings.**	Olustee, Fla.	No	
S5 2-45 (C)	Application of the oxo and related reactions to terpenes and resin acids to produce alcohols, aldehydes, and/or acids and characterization of the products thus obtained.**	Cincinnati, Ohio	Yes	8-B-2
S5 2-46 (C)	Synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers.	Ithaca, N. Y.	Yes	8-B-2
S5 2-48	Reaction of terpenes derived from turpentine with dienophiles to produce useful acids, aldehydes, amines, nitriles, sulfones, and related derivatives.**	Olustee, Fla.	Yes	8-B-1
S5 2-49 (C)	Investigation of the acid-catalyzed dimerization of alpha-pinene.	Gainesville,Fla.	Yes	8-B-1
S5 2-51	Development of practical methods for preparing the levopimaric acid-formaldehyde adduct, and the evaluation of this and selected derived products for industrial uses.**	Olustee, Fla.	Yes	8-B-1
S5 2-52	The preparation and reactions of epoxides and ozonization products of resin acids and their derivatives, to explore potential new industrial uses.	Olustee, Fla.	Yes	8-B-2

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S5 2-53	Development of improved polyester resins from resin acids.	Olustee, Fla.	Yes	8-B-2
S5 2-55	The preparation of polymerizable monomers for vinyl and condensation-type polymers from terpene acids and terpene acid derivatives.	Olustee, Fla.	Yes	8-B-1
S5 2-56	Conversion of rosin, resin acids and pine gum derivatives into polyols for use in polyurethane applications.*	Olustee, Fla.	Yes	8-B-1
S5 2-57	Production of useful chemicals from terpenes and resin acids by free radical addition of functional groups.*	Olustee, Fla.	Yes	8-B-1
S5 5-	New and Replacement Crops Utilization Investigations - Southern Region			
S5 5-51	Investigations of the chemical characteristics of new sweet sorghum canes which determine their suitability for sugar recovery.	Weslaco, Texas	Yes	9-A-1
SU-0-0-1 (DC)	Development of new and improved processed peach products, with special emphasis on the suitability of existing commercial varieties in the Southeast for the production of fresh peach concentrates.**	Experiment,Ga.	Yes	11-B-1
SU-0-0-2 (SG)	Improvement of fat emulsions suitable for use in intravenous alimentation.**	New Orleans, La.	Yes	2-D-1
SU-0-0-3 (AID)	A study of the preparation of cottonseed and peanut flours and their derived products for human consumption in developing countries.	New Orleans, La.	Yes	5-D-1
SU-0-0-4 (SG)	Development of cottonseed oil emulsions suitable for extended use in intravenous nutrition.*	New Orleans, La.	Yes	2-D-1
SU P 1	Seed Protein Pioneering Research Laboratory.***	New Orleans, La.	Yes	5-A-1
SU P 2	Plant Fibers Pioneering Research Laboratory.***	New Orleans, La.	Yes	1-A-4
UR-A7-(40)-3	A study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes.	Bombay, India	Yes	2-A-3
UR-A7-(20)-4	Investigation of the photochemical degradation of cotton to derive information which would enhance the utilization of cotton.	Bombay, India	Yes	1-A-3
UR-A7-(20)-19	A study of the relation between fine structure and mechanical properties of cotton fibers by swelling and stretching treatments, as a means of improving the properties, and thereby increasing the utilization of cotton.	Ahmedabad, India	Yes	1-A-4
UR-A7-(40)-26	Studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil, to provide possible new outlets for the utilization of cottonseed oil.	Bangalore, India	No	
UR-A7-(40)-28	Investigation of the synthesis and properties of new-type glycol monoalkyl ethers for the control of water evaporation, to extend the industrial utilization of cottonseed oil.	Poona, India	Yes	4-A-1

* Initiated during reporting year.
** Discontinued during reporting year.
*** There are no line projects under this work project.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
UR-A7-(20)-30	Investigation of new solvents for molecular weight determination of cellulose, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton.	Bombay, India	Yes	1-A-3
UR-A7-(20)-32	Investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics to provide basic information for the improvement of cotton products.	Bombay, India	Yes	1-A-3
UR-A7-(20)-33	Investigations of the preparation of radioresistant and radiosensitive celluloses to obtain basic information needed for useful applications of high energy radiation in cotton textile processing, thereby enhancing the utilization of cotton.	Bombay, India	No	
UR-A7-(20)-46	A study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber to obtain basic information needed to improve cotton processing and utilization.	Ahmedabad, India	Yes	1-A-1
UR-A7-(20)-51	Investigation of means to minimize fiber hooked ends in cotton card and drawing slivers to develop processing organizations of optimum efficiency, and thus to promote increased utilization of cotton.	Ahmedabad, India	Yes	1-B-5
UR-A7-(20)-59	An investigation of moisture sorption and desorption by crosslinked cotton celluloses over the entire humidity range, in relation to the state of swelling under which the cellulose is crosslinked and to other properties of the crosslinked celluloses, to obtain basic information of value in increasing the textile uses of cotton.	Delhi, India	No	
UR-A7-(20)-84	An investigation of heat and mass transfer rates and other basic engineering concepts as related to the drying and curing of resin-treated cotton textiles by counter-current solid-gas contact systems, to obtain fundamental information necessary to devise more efficient textile processing techniques, thereby increasing the utilization of cotton in textile applications.	Delhi, India	Yes	1-B-5
UR-A7-(20)-85	An investigation to determine the factors that affect the drafting capacity, optimum conditions, spinning efficiency, and yarn quality of the direct sliver spinning system to provide information needed to improve cotton processing and increase the utilization of cotton products.	Ahmedabad, India	No	
UR-A7-(20)-87	Investigations of the correlation between several important physical properties of woven cotton apparel fabrics and their performance in actual service tests, to obtain information needed for the improvement of cotton textiles.*	Delhi, India	No	
UR-A7-(20)-120	A study of factors affecting curling and bursting of preponderantly warp- and filling-faced cotton fabric structure during processing of cotton into end-use products.*	Bombay, India	No	
UR-A7-(20)-124	A study of the synthesis and properties of pure saturated diacid and triacid triglycerides for use as model compounds in obtaining basic information needed to improve the utilization of cottonseed oil.*	Bombay, India	No	
UR-A10-(40)-34	Investigation of π-complexed organometallic compounds derived from polyunsaturated fatty acids, to obtain fundamental information needed in expanding the utilization of cottonseed oil.	Haifa, Israel	Yes	4-A-1

* Initiated during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
UR-A10-(20)-50	A fundamental study of the oxidation of cotton and crosslinked cotton by hypochlorite, hypobromite, and other oxidizing agents, to obtain information needed on the kinetics of the oxidation and the changes in physical and chemical properties, in order to improve the characteristics of cotton for various end uses.	Jerusalem, Israel	Yes	1-B-3
UR-A10-(40)-52	Development of methods for the improved preparation of protein hydrolysates for the determination of amino acids, to provide a more accurate means for assessment of protein quality and nutritive value of oilseed proteins, thus contributing to their increased utilization.*	Jerusalem, Israel	No	
UR-A10-(40)-53	A study of the preparation of new chemical derivatives from acrylonitrile and fatty acids derived from the oils of cottonseed, tung, parsley seed, Limnanthes douglasii, Cuphea, and other oilseeds of the southern region of the United States to obtain information leading to potential new uses for these oils.	Jerusalem, Israel	No	
UR-A10-(40)-54	An investigation of metalation reactions employing alkali and alkaline earth metals and their derivatives for the modification of mono- and dienoic fatty acids to provide increased functionality, thereby leading to possible new uses for cottonseed, Limnanthes douglasii, and umbelliferous oils in industrial applications.	Jerusalem, Israel	No	
UR-A10-(20)-56	The synthesis and determination of the properties of new aziridinyl phosphorus compounds having potential for use in the treatment of cotton to afford new products of increased utility.	Jerusalem, Israel	No	
UR-A11-(10)-23	A study of the distribution of the major proteins of rice within subcellular particles, and the distribution of these particles in the cellular structure of the rice kernel, to obtain basic information needed for developing new and improved rice products and methods for producing them.*	Kyoto, Japan	No	
UR-A11-(40)-24	Studies of the biochemical mode of action of aflatoxins and their biodegradation, to obtain basic information needed for control of these toxins in cottonseed, peanuts, and other agricultural commodities that may be exposed to contamination by Aspergillus flavus.*	Anjo, Aichi, Japan	No	
UR-A11-(40)-29	An investigation of the chemical composition and reactivity of the nucleic acids of cottonseed, to obtain basic information needed for the increased utilization of this commodity.*	Kyoto, Japan	No	
UR-E4-(20)-1	A fundamental study of the nature and origin of reversals in cotton fibers, and of their relation to mechanical properties of these fibers, to obtain information needed in the development of improved cotton products.	Ghent, Belgium	Yes	1-A-2
UR-E9-(20)-61	A fundamental study of the relation of crystallinity to accessibility in native and modified cotton, to obtain information on the supermolecular structure of cotton that is needed in the development of improved cotton products.**	Paris, France	Yes	1-A-4
UR-E10-(20)-2	Development of an apparatus for counting neps in cotton card web as an aid toward increasing the quality of cotton products.	Reutlingen-Stuttgart, West Germany	Yes	1-A-7

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
UR-E15-(40)-33	Investigations on the physical and physicochemical properties of cottonseed proteins, to obtain basic information needed for the increased utilization of cottonseed.	Rome, Italy	Yes	2-A-1
UR-E15-(40)-35	A study of the mechanism of gossypol toxicity counteraction by L-lysine to gain information needed to permit the increased use of cottonseed products in animal feeds.	Milan, Italy	Yes	3-B-1
UR-E15-(40)-44	Experimental studies to elucidate the role of cottonseed meal in the induction of hepatoma in rainbow trout to obtain fundamental information concerning the suitability of cottonseed meal for use in rations for this species.	Aosta, Italy	Yes	3-B-1
UR-E19-(20)-4	A fundamental study of the role of the structural elements of the cotton fiber in response to stress in deformation and recovery, to obtain information needed in the development of improved cotton products.**	Delft, Holland	Yes	1-A-2
UR-E19-(20)-12	An investigation of the fundamental mechanisms and bonding forces that could be used to improve the tensile strength and other physical properties of cotton textiles, as a means of increasing the utilization of cotton.	Delft, Holland	Yes	1-A-2
UR-E21-(20)-27	An investigation of the mathematical and theoretical aspects of the relationship between the fiber length distribution of cotton specimens before and after sample preparation to obtain basic information needed to improve cotton processing.	Lodz, Poland	Yes	1-A-7
UR-E25-(40)-19	An investigation of the rate of reaction of protein with carbohydrates in peanuts, to provide information leading to improved peanut products, thereby increasing the utilization of this commodity.*	Granada, Spain	No	
UR-E25-(20)-31	A study of the measurement of the "total hairiness" of cotton yarn and the determination of the mechanical factors contributing toward its formation, to obtain basic information needed to improve the processing of cotton into textiles.	Barcelona, Spain	Yes	1-A-5
UR-E25-(50)-36	Development of new or improved methods of synthesizing, isolating, and purifying selected terpene alcohols for use as standards, to obtain basic information on the composition and properties of products made from pine gum as an aid in developing new industrial uses for naval stores products.	Barcelona, Spain	Yes	8-A-1
UR-E25-(20)-42	An investigation of the effect of fiber properties on drafting tenacity during spinning of cotton and the interrelationships between fiber properties, drafting tenacity, yarn properties, and end breakage, to obtain basic information related to processing properties in the utilization of cotton.*	Barcelona, Spain	No	
UR-E26-(20)-1	Investigation of the mechanism of crease formation and recovery in ease-of-care treated cotton fabrics to supply fundamental knowledge required for the design of improved textiles, thereby increasing the utilization of cotton.	Gothenburg, Sweden	No	
UR-E26-(20)-2	Fundamental investigation of setting reactions for cotton fabrics and garments, to develop information basic to the improvement of cotton products, thereby increasing the utilization of cotton.	Gothenburg, Sweden	Yes	1-A-2

* Initiated during reporting year.
** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
UR-E26-(20)-6	Basic investigation of the behavior of cotton subjected to aerodynamic forces, for the purpose of improving the processing characteristics of cotton textiles.	Gothenburg, Sweden	Yes	1-B-5
UR-E27-(20)-2	A study of the chemistry and structural nature of the bonds formed between formaldehyde and cellulose in formaldehyde-treated cottons to provide basic information needed to improve the utility of the cotton fabrics.	Zurich, Switzerland	Yes	1-A-4
UR-E29-(40)-26	Studies on the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components, to obtain fundamental information that will contribute to the development of improved edible products and hence to expanded utilization of cottonseed oil.**	Leatherhead, Surrey, England	Yes	2-A-3
UR-E29-(20)-55	A fundamental study of the preparation and properties of phosphazene (phosphonitrilic) and phosphoryl chloride derivatives having potential for reaction with cotton cellulose, to obtain information needed in the development of new useful products from cotton, thus increasing its utilization.	London, England	Yes	1-B-1
UR-E29-(20)-65	A study of the effect of caustic soda and other swelling agents on the fine structure of cotton, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton.	Manchester, England	Yes	1-A-4
UR-E29-(20)-78	Investigation of chemical modifications of cotton fabrics involving control of lateral molecular order and distribution of crosslinks, to provide basic information needed to improve the performance characteristics of cotton fabrics as a means of increasing their utilization.	Manchester, England	No	
UR-01-(40)-2	An investigation of the chemistry and biological effects of cyclopropenoid compounds that occur in cottonseed and cottonseed products, to obtain basic information needed to improve the utilization of these commodities.	Ryde, New South Wales, Australia	Yes	2-A-3
UR-S9-(40)-2	Preparation, characterization, and evaluation of derivatives of gossypol from cottonseed for use as biologically active materials, ultraviolet adsorbers and other valuable products.**	Montevideo, Uruguay	Yes	4-A-1

** Discontinued during reporting year.